PB87-165452

Exploration, Development, and Production of Crude Oil and Natural Gas (Technical Report) Appendix E. Role and Function of EPA (Environmental Protection Agency) Sample Control Center

(U.S.) Environmental Protection Agency
Washington, DC

31 Jan 87

U.S. Department of Commerce
National Technical Information Service
NTIS

United States
Environmental
Protection
Agency

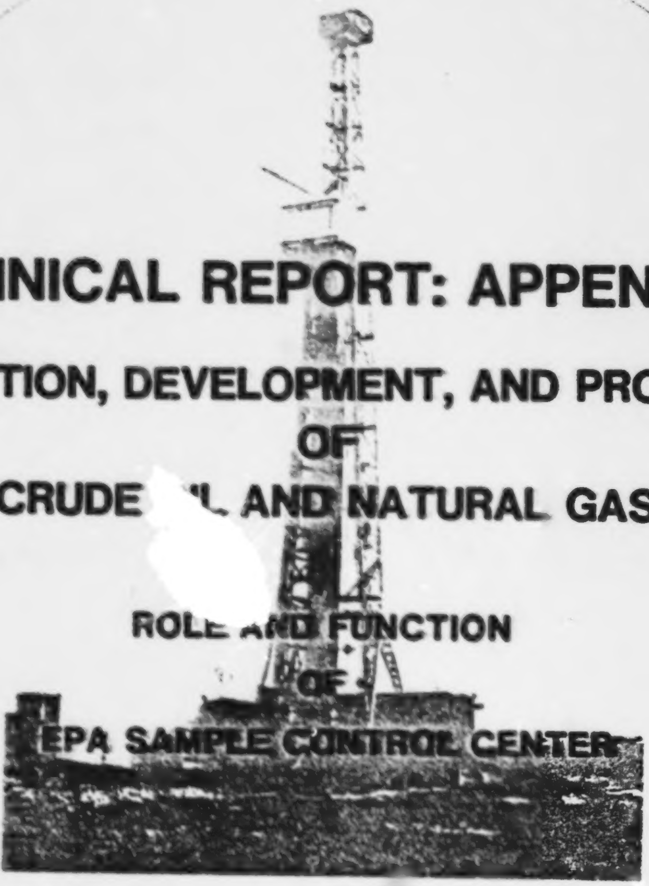
Office of Water
Regulations and
Standards (WH-552)
Washington, D.C.
20460

Office of Solid Waste
and Emergency
Response
Washington, D.C.
20460

EPA
530-SW-87-005 E

PB87-165452

EPA



TECHNICAL REPORT: APPENDIX E

EXPLORATION, DEVELOPMENT, AND PRODUCTION
OF
CRUDE [illegible]L AND NATURAL GAS

ROLE AND FUNCTION
OF
EPA SAMPLE CONTROL CENTER

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The Industrial Technology Division (ITD) of EPA has created the Sample Control Center to ensure the orderly flow of samples and analytical data through the sampling, analysis and quality assurance system. This appendix gives the purposes and objectives of the Sample Control Center and an overview of the operations conducted by the Center.

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APPENDIX E

ROLE AND FUNCTION OF THE EPA SAMPLE CONTROL CENTER

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U.S ENVIRONMENTAL PROTECTION AGENCY
INDUSTRIAL TECHNOLOGY DIVISION
SAMPLE CONTROL CENTER

An Introduction to the Sample Control Center
and a
Guide to Its Services

INTRODUCTION

The Industrial Technology Division (ITD) (formerly the Effluent Guidelines Division) of EPA conducts programs for collection and analysis of samples from treatment systems in industrial plants. The objective of this sampling and analysis effort is to determine the presence and to quantify the levels of pollutants so that ITD can promulgate nationwide standards and guidelines for control of industrial discharges. Personnel from EPA Headquarters, EPA Regions, and independent contractors have been organized to collect and analyze samples on an as-needed basis by ITD.

The program conducted by ITD generates thousands of sub-samples and extracts for analysis. In order for analytical results to be valid, it is imperative that the collection, analysis, data collection, and data quality assurance processes be conducted in accordance with a carefully controlled schedule. It presently costs nearly $4,000 to collect, analyze, and quality assure the results from each sample. Any of the following problems can cause the data from a sample to be rendered useless and to necessitate repeating the process and expenditure:

loss of a sample
a delay in processing
unlabeled or mis-labeled containers
lack of control in tracking
violation of the sample holding time
improperly conducted analysis
failure to quality assure the result

ITD has created the Sample Control Center to ensure the orderly flow of samples and analytical data through the sampling, analysis, and quality assurance system, and to insure that the above problems are minimized.

This guide gives the purposes and objectives of the Sample Control Center, and an overview of the operations conducted by the Center. For the most part, these operations are controlled by forms. Throughout this guide, the names of these forms are shown in **boldface** type. Where appropriate, supporting documents are included as attachments to this guide. These attachments provide the details of the requirements for operation of the sample control, data collection, and quality assurance functions that the Center performs.

PURPOSE OF THE SAMPLE CONTROL CENTER

The Sample Control Center was conceived and designed to provide an efficient mechanism for controlling the logistics of the nationwide sampling and analysis programs for ITD. Practices and procedures have been implemented which control sample flow from collection through analysis and data flow through quality assurance and final reporting. Much effort has gone into making sure that the amount of control is sufficient to prevent sample and data misidentification or loss without the record-keeping becoming self-defeating.

Record-keeping is implemented through the Episode File; sample flow is controlled through Traffic Reports; analyses are controlled through Laboratory Chronicles, and data flow and data quality assurance are controlled through the Data Tracking and Quality Assurance systems.

The purpose of the Center is to maintain an orderly flow of samples, analyses, and data, so that ITD objectives are met. The management of the Center has made every effort to accomplish these purposes by thoroughly studying the requirements of ITD Management, ITD Project Officers, EPA Regional Surveillance and Analysis personnel, ITD sampling contractors and analysis laboratories, and by implementing a system for reporting and quality assuring gas chromatography-mass spectrometry data recieved on magnetic tape.

OBJECTIVES OF THE SAMPLE CONTROL CENTER

The objectives of the ITD Sample Control Center are to:

Provide logistical support to everyone involved in the field operation of the sampling and analysis program.

Insure effective traffic control of all materials.

Insure data integrity.

Provide status of all samples, extracts, and data.

Consolidate and report management information and analysis results to ITD.

Provide statements of data quality to ITD Project Managers.

Increase the cost efficiency of sampling and analysis programs.

Provide consistent, time-saving procedures for Sampling Contractors, Regional S&A personnel, EPA Laboratories, and Contract laboratories.

OVERVIEW OF SAMPLING, ANALYSIS, AND QUALITY ASSURANCE PROCEDURES

The Sample Control Center is brought into action when an ITD Project Officer has need for analysis data to support development of an Effluent Guideline. The request for analysis generally comes through the ITD Project Officer responsible for operation of the Center. This Project Officer is presently W A Telliard. Schedules, laboratory availability, level of sampling effort, and funding mechanisms are worked out with the Project Officer, and the sampling and analysis effort can then begin.

To begin the scheduling process, SCC personnel create a permanent Episode Sample File containing the following scheduling information:

Name and affiliation of the individual requesting the analysis.
The specific analyses requested.
Date of the request.
Responsible ITD Project Officer.
Facility name and location.
Industrial Category.
Scheduled sampling and shipping dates.
Sampling organization, contact, and telephone number.
Number and types of samples to be collected.

The following sub-sections give details of the Sampling, Analysis, Data Reporting and Quality Assurance activities of the Sample Control Center which support Project Officers within the Industrial Technology Division of EPA.

SAMPLING

When an ITD Project Officer initiates a sampling effort for a particular location, the Sample Control Center schedules these samples for shipment to a particular Analysis Laboratory, opens a file on that sampling Episode, and prepares a packet of material to send to the Sampler. (An Episode refers to a group of samples collected at a given plant or location within the same calendar week.) All forms and labels bear a single identification number which serve as a permanent reference to collection and analysis of samples at a given sampling point. The packet normally contains the following:

Episode Number assignments
Laboratory assignments
Sample Volume and Preservative Requirements for special analytes
Traffic Report(s) - With Adhesive Sample Labels

The above items were designed to:
be easy to use
keep writing requirements to a minimum
provide clear guidance and instruction
allow for individualized training
be amended, modified as needed
provide a documented record of events

The **Traffic Report** is a four-part carbonless form used primarily to document the dates samples were taken, shipped and received. The top white copy is returned by the Sampler to SCC. The yellow copy becomes the Sampler's copy. The bottom two copies are then sent in a Ziploc bag with the samples to the Analysis Laboratory. Upon receipt of the samples, the Analysis Laboratory completes the required information on the Traffic report, returns the pink copy promptly to the Center, and retains the gold copy for its records. There are four types of Traffic Reports currently in use by the center:

Organics Traffic Report

Metals Traffic Report

Asbestos Traffic Report

Generic Traffic Report

The color coding of the copies is alike in all four types and the information recorded on each is very similar. An example of an **Organics Traffic Report** is shown in Attachment 1. This example does not show the color coding or that the sample numbers are adhesive backed. Personnel who wish to become familiar with these forms should request a copy from the Center. The organics, metals, asbestos, and generic forms were designed to accommodate the differences in the sampling and analysis programs.

The preprinted and prenumbered Adhesive Sample Labels affixed to the Traffic Reports are secured to sample containers by the Sampler. Where necessary, the label is protected from attack by water and solvents with clear cellophane tape which is designed to resist this attack.

The Sampler uses one Traffic Report per day for all samples in a sampling episode, if possible. If not, more Traffic Reports are used, as required. If organics and metals samples are collected from the same point, the Sampler assures that the Organics and Metals Traffic Reports have the same identification number. A quantity of labels printed with numbers only are provided so that the Sampler may describe and identify any additional samples taken. After sampling, the first page (white copy) of the Traffic Report is mailed to the Sample Control Center, the yellow copy is retained by the Sampler, and the remaining two copies are sent to the Analysis Laboratory with the samples. The Sample Control Center typically includes two extras.

When Laboratory Chronicles, Additional Sample Labels, and/or Description of Metals Analysis Sheets are included in the packet, they are sealed in Ziploc bags and forwarded to the lab(s) with the first sample shipment.

ANALYSIS

Laboratories analyze samples under analytical contracts with EPA or under Special Analytical Services (SAS) contracts with the Sample Control Center through Viar and Company. These contracts specify requirements for sample analysis including the analytical methods. The **Description of Metals Analysis Sheet** identifies the elements to be analyzed by the metals laboratory. The Center sends the Description(s) to the lab(s) prior to the first shipment of metals samples. An example of this form is shown in Attachment 2.

The Sample Control Center interacts in the chemical analysis of samples only when the laboratory calls with an analytical problem. In the past, most of these problems have dealt with the analysis of difficult organic samples. The latest revisions of the methods for analysis of organics contain provisions for analysis of such difficult samples, so the number of such calls have been minimized. Nevertheless, in the event that a laboratory calls with a problem related to the analysis, and Center personnel cannot answer the question, the Project Officer is contacted immediately for resolution. Delays in responding to a laboratory's request for resolution of a problem can delay the analysis and submission of results.

Volatile Gases and Water Soluble Compounds. EPA Method 1624 permits analysis of certain "volatile gases and water soluble compounds" at one of two concentrations. IFB and SAS contracts require the laboratory to notify the Center at which concentration these compounds will be calibrated and analyzed. The **Method 1624 - Volatiles, Volatile Gases and Water Soluble Compounds Standards' Concentrations** form is used for this purpose. An example of this form is shown in Attachment 3. This form must be received by the Center, and the concentrations loaded into the appropriate table in the computer prior to the time data on tape are received from the laboratory; otherwise, certain computerized tests of the data submitted by the laboratories will be failed.

Sample Logistics. Laboratories are required to submit data related to sample logistics. Each laboratory notes on its copies of the **Traffic Report** the dates the samples were received and their condition on receipt. The pink copy is returned to the Sample Control Center and the gold copy is retained by the lab.

During organic sample preparation and analysis, dates, technicians' names and any problems or relevant comments are recorded on the **Laboratory Chronicle**, shown in attachment 4. Upon completion of analysis, the white copy of the Laboratory Chronicle is included as part of the data package and the yellow copy is retained by the lab. The laboratory is responsible for the analysis and for full compliance with all specifications in the contract, including those in the quality assurance section of the analytical method.

DATA REPORTING

Data reporting requirements are project specific, but follow a general pattern. Data related to sampling and sample tracking are stored in the SAMple TRACking (SAMTRAC) system. Data from the analysis of organics are reported on magnetic tape according to a set of documents which define the tape specifications and format; all other data (metals, asbestos, and other special analyses) are reported on data sheets.

Sampling Data. Data from the Project Officer, Traffic Reports and Laboratory Chronicles related to sampling and sample tracking are entered into the SAMTRAC system at the Sample Control Center. These data are accumulated in the Episode Sample File. From this file, various reports related to the sampling data can be produced. One such report related to the sampling and analysis of organics is the **Isotope Dilution Episode Sample Report**. An up-to-the-minute copy of this report is maintained at the Sample Control Center. From this report, all plant and sample specific information can be retrieved. A copy of a page from this report shown in Attachment 5. The sample stream descriptions and comments listed on this report are explained more fully in the Data Element Dictionary.

Organic Data. Prior to 1983, organic data were received on data sheets. In 1983 ITD began receiving these data on magnetic tape. The documents which define the tape specifications and format are the:

Data Element Dictionary (ITD Data Elements)
Analytical Methods (presently Methods 1624C and 1625C)
Instructions for Use of Labeled Compounds
[Specifications for] Quantitation Reports on Magnetic Tape

The present revisions of these documents are Attachments 6 thru 9, respectively.

Data Element Dictionary. The **ITD Data Elements** define the data elements collected and stored in the data base at EPA's National Computer Center (NCC). A "Summary of Data Sources for Collection of Organic Priority Pollutant Data" is given in the table on pages 6 and 7 of the ITD Data Elements in Attachment 6. This table shows the source of each data element. Notice that the Sample Number is common to all data sources; i.e., the Sample Number ties all of the data together.

Analytical Methods. The **Analytical Methods** are instructions that laboratories are to follow when they analyze the samples. Methods 1624 and 1625 are isotope dilution GCMS methods for analysis of organic pollutants. These methods are specified in Invitation for Bid (IFB), Special Analytical Services (SAS), and other contracts. Methods 1624 and 1625 are shown in Attachment 7.

Instructions for Use of Labeled Compounds. The analysis of organic compounds by isotope dilution requires the use of stable (non-radioactive) isotopically labeled compounds. The **Instructions for Use of Labeled Compounds** is included in the contract documents, and specifies how the labeled compounds are to be mixed and spiked into the samples, standards, and blanks. These instructions are shown in Attachment 8. The Center supplies labeled compound solutions on an "as needed" basis by the laboratories. Labeled compound solutions have been synthesized to date by Merck & Co., Isotopes Division, Quebec, Canada. These solutions are kept in freezers at the SCC Chemical Repository in Baltimore, and are shipped as directed by the Center personnel.

After the laboratory completes analysis of a sample or group of samples, the lab is required to ship data and other deliverables to the Center, and to other locations specified in the contract or as directed by the Project Officer.

Quantitation Reports on Magnetic Tape. Organics data are shipped to the Center on magnetic tape per the specifications in **Quantitation Reports on Magnetic Tape** in Attachment 9. Tapes are received by the Center, logged-in, and shipped to the NCC in Research Triangle Park, North Carolina. Data are deblocked and quality assured as described in the section on "Quality Assurance" below.

Sample Extracts. Other deliverables required by the organics' contracts include sample extracts and raw GCMS data on magnetic tape. Sample Extracts are normally shipped to the Repository where they are stored in freezers for later use. A **Sample Extract Transmittal Form** is shown in the contract and accompanies each shipment of extracts. Requirements for shipment of sample extracts are shown in Attachment 10.

Raw GCMS Data Tapes. Raw GCMS Data Tapes (not to be confused with tapes containing Quantitation Reports) are normally shipped to Dr John McGuire at EPA's Environmental Research Laboratory in Athens, Georgia. Specifications for raw data tapes are contained in Attachment 11. A **Tape Transmittal Form**, also shown in the Attachment, accompanies each tape. The extracts and tapes are shipped as directed by the Project Officer responsible for operation of the Center.

Metals Data. Metals sampling, analysis, and data reporting are similar to those given above for organic samples; i.e., there are contracts and methods for analysis of metals and forms for tracking and reporting. Metals data are reported on data sheets using the Sample Control Center **Laboratory Results, Metals Analysis form**. A copy of this form is shown in Attachment 12, and is included in metals' contracts. The Project Officer specifies the metals to be analyzed in each sample, and the technique to be used.

QUALITY ASSURANCE

The analysis laboratory and the Sample Control Center quality assure the data received at the Center. All data are to be quality assured by the analytical laboratories prior to submittal, so the Center's main function is to check that the laboratory has properly assured the data.

GCMS Data. GCMS data are received in the form of quantitation reports on magnetic tape and are 100 percent quality assured by the Center. A **Quantitation Report Tape Transmittal Form** accompanies each tape and lists the specific files on that tape. This form is shown at the back of Attachment 9. When a tape is received at the Center, it is assigned a tape number and a record is entered in the tape log. The tape is then forwarded to NCC for processing. The processing steps are shown in the **SCC Tape Processing Steps** document in Attachment 13. The quality assurance process occurs in four major steps:

(1) Tapes are "de-blocked" into individual files. Each file represents a quantitation report or a piece of a quantitation report for a given sample, standard, or blank. There can be at most three pieces (files) which make up a full quantitation report. And because each sample is divided into three fractions (acid, base/neutral, and volatile), up to nine files may represent the results of analysis of a single sample. The de-block report gives errors related to sample and analysis logistics. The data elements tested are:

The date and time analyzed. This date and time are compared with the date and time sampled to assure that the holding time for the sample (or extract) has not been exceeded.

An instrument identifier. All data are tracked by each specific GCMS instrument within a laboratory to certain that the instrument is in calibration when samples are analyzed.

The shift on which the sample was analyzed.

The quantitation report type. This element identifies the analysis as a Sample, Standard, or Blank.

The EPA Sample Number, or the concentration of the standard.

A single letter code for the fraction.

The concentration/dilution factor (see the Data Element Dictionary for a detailed definition of this data element).

The date and shift on which the sample was extracted, if applicable.

A standard and blank are required for each set of samples started through the extraction process on a given shift.

If any of these data elements are missing or incorrect in any file, that file cannot be processed further until the error is resolved with the lab. or the proper information deduced from the Tape Transmittal Form.

(2) Each file is submitted for a Level 1 Edit. This edit tests all data elements specific to a complete quantitation report. Pieces of a quantitation report are merged for this process. Data are tested against specifications in Methods 1624 and 1625. Technical data elements tested are the:

Compound number.

Compound reference number

Quantitation m/z. Scan number.

Retention time.

Reference compound.

Relative retention time.

Amount.

Unit of measure.

Response factor.

In addition, reference (library) information is tested. Data elements tested are the:

Library retention time.

Library relative retention time.

Library amount.

Library response factor.

All of the data elements above (technical and library) are literally tested; i.e., the data fields requiring a number only are tested to determine if non-numeric characters are present in the field (including alphabetical characters, symbols, hyphens, etc). The specific tests at Level 1 are given in greater detail in Attachment 14.

A Level 1 Edit Report is produced which shows errors, if any. If errors are present which will prevent the Level 2 Edit (described below) from running, they are resolved with the lab before the files are submitted for Level 2 Edit.

(3) The Level 2 Method Edit verifies the presence and order of quality assurance sample and field sample analyses.

(4) The Level 2 Final Edit tests all quantitation reports on a tape against calibration and other data stored for the instrument on which the samples were analyzed, to verify that the instrument remained in calibration for all analyses performed. All specifications requiring multiple

GCMS runs are tested by this edit; for example, if a tape contains calibration data, calibration linearity over the 5 point calibration is tested.

In addition to reporting results on magnetic tape, Report Form 1, which is shown in the specifications for "Quantitation Reports on Magnetic Tape" may be used by the laboratory for reporting results of hard to analyze compounds. These results supercede data on tape.

After all tests are passed, and open data items are resolved with the laboratory, data are merged into a final data file. This file is processed through the "Data Base Print' program to produce a final report for the Project Officer. This report lists the results of analyses of all compounds in all samples, and includes selection of the result at the most dilute level within the calibration range of the GCMS instrument. Statements of Data Quality are produced for labeled compound recoveries from samples and for analysis of standards by the laboratory. These statements are consistent with those required by the Methods.

CONCLUSIONS

The Sample Control Center is currently managing the collection and analysis of samples for numerous ITD programs. Among these are Organics Screening and Verification, Metals, Asbestos, and Residual Waste. Since the inception of the Center, all interested parties have benefited from cost efficiency, immediate problem response, rapid data turnaround and access to computerized sample tracking information. In summary, the effectiveness of the Sample Control Center as a management tool has been proven.

The success of the control system is based on the consistency of procedures surrounding the use of the reporting forms, the extensive computerized and manual record-keeping and the constant phone and written communication among Center, Sampling, Analysis and EPA personnel.

Should you have any questions about the operations of the Sample Control Center or if you would be interested in receiving copies of any of our reporting instruments, please contact us at (703) 557-5040. Center personnel would be pleased to be of assistance.

ATTACHMENT 1

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USEPA INDUSTRIAL TECHNOLOGY DIVISION
SAMPLE CONTROL CENTER
P.O. BOX 1407
ALEXANDRIA, VA 22313
703/557-5040 / FTS-8-557-5040

EPISODE NO:

RANGE OF SAMPLE NOS: —

ORGANICS TRAFFIC REPORT

INDUSTRIAL FIRM SAMPLED: ______________________

CITY: ______________________

STATE: ______________________

INDUSTRIAL CATEGORY: ______________________

CONFIDENTIAL: ____ YES ____ NO

SAMPLING OFFICE: ______________________

SAMPLER: ______________________

SAMPLING DATE

BEGIN: ______________________

END: ______________________

SHIPPING INFORMATION

SHIP TO: ______________________

ATTN: ______________________

CARRIER: ______________________

AIRBILL NO: ______________________

DATE SHIPPED: ______________________

[redacted]

	SAMPLE POINT DESCRIPTION								SAMPLE TYPE										
SAMPLE NUMBER	RAW WATER (CITY, RIVER, WELL)	IN LINE PROCESS	UNTREATED EFFLUENT (RAW WASTE WATER)	TREATED EFFLUENT	OTHER (SPECIFY)	ADDITIONAL SAMPLE DESCRIPTION	PH LEVEL	SAMPLE POINT FLOW UNITS: ____	C COMPOSITE (EXTRACTABLE ORGANICS)	CB COMPOSITE BLANK	G GRAB (EXTRACTABLE ORGANICS)	V1 VOA PRESERVED	V2 VOA PRESERVED (DUPLICATE)	V3 VOA UNPRESERVED	V4 VOA UNPRESERVED (DUPLICATE)	VB1 VOA TRIP BLANK	VB2 VOA TRIP BLANK ((DUPLICATE)	OTHER SPECIFY:

WHITE — SCC COPY   YELLOW — SAMPLER COPY   PINK — LAB COPY FOR RETURN TO SCC   GOLD — LAB COPY

ATTACHMENT 2

DESCRIPTION OF METALS ANALYSIS

Lab Name ____________________

No. of Samples Scheduled __________

Sampling Points ____________________

Industrial Category ____________________

Industrial Subcategory ____________________

Sample Numbers ____________ - ____________

Please analyze these metals samples as indicated below.

☐ ICP EMISSION (Elements 1-21) All 21

1. Calcium
2. Magnesium
3. Sodium
4. Aluminum
5. Manganese
6. Lead
7. Vanadium
8. Boron
9. Barium
10. Beryllium
11. Cadmium
12. Molybdenum
13. Tin
14. Yttrium
15. Cobalt
16. Chromium
17. Copper
18. Iron
19. Nickel
20. Titanium
21. Zinc

☐ FURNACE AA (Elements 1 - 6) Check specific elements

___ 1. Selenium
___ 2. Thallium
___ 3. Silver
___ 4. Arsenic
___ 5. Antimony
___ 6. Mercury

☐ Flame Atomic Absorption (Elements 1 - 20) Check specific elements

___ 1. Calcium
___ 2. Magnesium
___ 3. Sodium
___ 4. Aluminum
___ 5. Manganese
___ 6. Lead
___ 7. Vanadium
___ 8. Boron
___ 9. Barium
___ 10. Beryllium
___ 11. Cadmium
___ 12. Molybdenum
___ 13. Tin
___ 14. Cobalt
___ 15. Chromium
___ 16. Copper
___ 17. Iron
___ 18. Nickel
___ 19. Titanium
___ 20. Zinc

ATTACHMENT 3

METHOD 1624 A - VOLATILES
VOLATILE GASES AND WATER SOLUBLE COMPOUNDS
STANDARDS' CONCENTRATIONS

LAB # ____ LAB NAME ______________________________

LABELED COMPOUND SPIKING SOLUTION CONCENTRATIONS	
COMPOUND	CONCENTRATION UG/L
203 - ACRYLONITRILE-D3	
216 - CHLOROETHANE-D5	
245 - CHLOROMETHANE-D3	
246 - BROMOMETHANE-D3	
288 - VINYL-D3 CHLORIDE	
614 - 2-BUTANONE-4,4,4-D3 (MEK)	
615 - DIETHYL-D10 ETHER	
616 - ACETONE-D6	

PRIORITY POLLUTANT CALIBRATION CONCENTRATIONS					
	CONCENTRATION UG/L				
COMPOUND	10	20	50	100	200
516 ACETONE					
002 ACROLEIN					
003 ACRYLONITRILE					
046 BROMOMETHANE					
016 CHLOROETHANE					
045 CHLOROMETHANE					
515 DIETHYL ETHER					
514 METHYL ETHYLKETONE					
088 VINYL CHLORIDE					

ATTACHMENT 4

U.S. EPA INDUSTRIAL TECHNOLOGY DIVISION SAMPLE CONTROL CENTER

ORGANICS SUPERSCREENING LAB CHRONICLE

Sample No.

Lab Name ______________________ Industrial Category ______________________

SAMPLE DESCRIPTION	Technician Name Date Extracted	Analyst Name Date Analyzed	Detail below pertinent information and/or problems associated with sample preparation and analysis and the measures taken to overcome those problems. Use supplementary sheets to continue if necessary.
EXTRACTABLE (BASE/NEUTRAL)			
EXTRACTABLE (ACID)			
EXTRACTABLE (PESTICIDES)			
COMPOSITE BLANK (BASE/NEUTRAL)			
COMPOSITE BLANK (ACID)			
COMPOSITE BLANK (PESTICIDES)			
VOA (Preserved ___ Unpreserved ___)	N/A		
VOA TRIP BLANK	N/A		

Use section below to describe QA/QC samples if received.

QA/QC EXTRACTABLE (BASE/NEUTRAL)			
QA/QC EXTRACTABLE (ACID)			
QA/QC EXTRACTABLE (PESTICIDES)			
QA/QC VOA (Preserved ___ Unpreserved ___)	N/A		
QA/QC EXTRACTABLE (BASE/NEUTRAL)			
QA/QC EXTRACTABLE (ACID)			
QA/QC EXTRACTABLE (PESTICIDES)			
QA/QC VOA (Preserved ___ Unpreserved ___)	N/A		

— ITD SAMPLE CONTROL CENTER COPY —

ATTACHMENT 5

```
PART 2 : EPISODE/SAMPLE
-----------------------

 EPISODE SAMPLE                                                                                            NO OF
 NUMBER  NUMBER    TYPE     TYPE DESCRIPTION        SITE   SITE DESCRIPTION       LAB ID    LAB NAME     SAMPLES
 ------- ------    ----     ---------------------   ----   --------------------   --------  ---------    -------

 1061                                                                                                      01

         15056       EP     EPA SAMPLE               C     (INF)-UNTREATED EFF
              COMMENT 1 :   S376   BRINE
            SAMPLE DATE :   062086
                     PH :   08.000

         15056       TB     VOA TRIP BLANK           H     (OTH)-OTHER
            SAMPLE DATE :   062086
                     PH :   08.000

                                    ACTUAL SAMPLE COUNT :   2

 1062                                                                                                      02

         15057       EP     EPA SAMPLE               H     (OTH)-OTHER
              COMMENT 1 :   S380   PIT CONTENTS
            SAMPLE DATE :   062386
                     PH :   07.000

         15057       TB     VOA TRIP BLANK           H     (OTH)-OTHER
            SAMPLE DATE :   062386

         15058       EP     EPA SAMPLE               H     (OTH)-OTHER
              COMMENT 1 :   S380   PIT CONTENTS
              COMMENT 2 :   S302   SLUDGE
            SAMPLE DATE :   062386
                     PH :   07.000

                                    ACTUAL SAMPLE COUNT :   3

 1063                                                                                                      01

         15059       EP     EPA SAMPLE               A     (SUP)-RAW WATER (SU
              COMMENT 1 :   S376   BRINE
            SAMPLE DATE :   062486
                     PH :   07.000

         15059       TB     VOA TRIP BLANK           H     (OTH)-OTHER
            SAMPLE DATE :   062486

                                    ACTUAL SAMPLE COUNT :   2

 1064                                                                                                      02

         15060       EP     EPA SAMPLE               H     (OTH)-OTHER
              COMMENT 1 :   S380   PIT CONTENTS
```

ATTACHMENT 6

ITD DATA ELEMENT DICTIONARY

NOTE: Pay special attention to the Concentration/Dilution Factor

ITD Data Element Dictionary

Following is a brief description of each data element which is to be stored in the ITD data base. The originating source for the data element is also given. The complete specification for each data element and an example of its use are given on a separate page following this summary.

Amount — The quantitative measurement of the compound determined by GC or GC/MS analysis. The amount is computed for the compound using the referenced internal standard or isotopic diluent and is multiplied by the concentration or dilution factor to yield final solution concentrations in ug/L.

Amount (Library) — The reference amount in the standard and the amount on which quantitation is based.

Bottle Number — A numeric code which uniquely identifies the bottles used for a particular sample.

Carrier Gas Flow Rate — The volumetric (volume/time) rate of flow of the carrier gas in the gas chromatograph, or the linear gas velocity (distance/time) when a capillary column is used.

Column Final Hold — The final temperature of the gas chromatograph column and the length of time that it was held.

Column Initial Hold — The initial temperature of the gas chromatograph column and the length of time that it was held.

Column Inside Diameter — The internal diameter of the gas chromatograph column.

Column Length — The length of the gas chromatograph column.

Column Temperature Program — The change in column temperature with respect to time giving the initial and final column temperatures.

Compound Comment Code — A coded value for any optional text that may be associated with each compound.

Compound Name — The name of the compound determined. The compound name corresponds to the EGD compound number, as given in the "Industrial Technology Division Compound Code Listing."

Compound Number — A numerical code which uniquely identifies each unique chemical compound, as given in the "Industrial Technology Division Compound Code Listing."

Compound Order Number — A numerical code that establishes the order of compound determination by the GC/MS. The code is used on the Quantitation Report to match segments of the compound data within the report.

Compound Type — A coded value which identifies a chemical compound as a priority pollutant (P), or surrogate (S), internal standard (I), or isotopic dilutent (D).

Concentration/Dilution Factor — The ratio of the volume of sample extracted or diluted to the volume analyzed.

Date Analyzed — The date that the sample fraction was analyzed by the laboratory.

Date Extracted — The date that the laboratory extracted the sample for analysis.

Date Sampled — The date the sample was taken by the field sampler.

Episode Comment Code — A coded value for comments associated with an episode.

Episode Number — The SCC assigned identification code with designates the specific sampling trip.

Fraction — A coded value which designates the compound as either an acid, base/neutral, volatile, pesticide or dioxin.

Fraction Comment Code — A coded value for any optional text that may be associated with each fraction.

Industrial Category Code — The classification of the industrial processes performed by the plant where a sample was taken.

Instrument — A coded value assigned by the laboratory that uniquely identifies each GC/MS instrument within a laboratory. All Calibration, Precision and Recovery, Standards and Blank Quantitation files will be tracked by this instrument number within Laboratory. Changing of this instrument number by the laboratory would necessitate the submittal of new calibration and other initial QA runs by the laboratory.

Laboratory — A numerical code used to identify the specific laboratory where the sample was analyzed.

Mass to Charge Ratio — Designates the quantitation ion. Abreviated as M/Z or M/E.

Method — A coded value which uniquely identifies the method protocol that was followed during analysis.

Peak Area — The area beneath the peak of a mass chromatogram. The peak area is proportional to the amount of the detected compound at an observed mass to charge ratio. It is used to compute the concentration of the compound present in the sample.

PH Level — The negative logarithm of the effective hydrogen ion concentration as expressed in grain equivalents per liter.

Plant Code — A numerical code used to distinguish specific plants which have been sampled.

Proprietary Indicator — A coded value which designates whether or not the analysis data from a sample is proprietary. Also indicates that confidentiality papers have been signed.

Quantitation Report Type — A coded value that uniquely identifies the particular type of quantitation report that is being submitted.

Reference Compound — A numeric code that is used as a pointer to the internal standard or isotopic diluent within a quantitation report.

Relative Retention Time — The quotient of the retention time of a compound divided by its internal standard or isotopic diluent.

Relative Retention Time (Library) — The relative retention time stored in the library. The value is based on the analysis of a standard containing both compounds.

Response Factor — The ratio between the response for the sample and a response for a standard under identical analytical conditions.

Response Factor (Library) — The response factor stored in the library. The value is determined from analysis of a standard.

Retention Time — The time it takes the identified compound to elute from the gas chromatograph.

Retention Time (Library) — The known time it takes an identified compound to elute from the gas chromatograph. The time is determined from analysis of a standard.

Sample Comment Code — A coded value for any optional text that may be associated with each sample.

Sample Number — The SCC assigned identification code which identifies the individual samples.

Sample Point (Site) — The specific point within an industrial wastestream where a sample was taken.

Sample Point Flow — The flow rate at the point at which the sample was taken. Value is recorded from a flow meter or other flow measuring device.

Sample Type — A coded value which describes the type of sample.

Scan Number — Gives the scan at which the compound was detected by the mass spectrometer.

Shift — The scheduled period of operation of the GC/MS instrument. Operation is divided into three shifts/day.

Time Analyzed — The time that the sample fraction was analyzed by the laboratory.

Unit of Measure — The unit of measurement for the amount.

The following chart shows the source of each data element.

SUMMARY OF DATA SOURCES FOR COLLECTION OF ORGANIC PRIORITY POLLUTANT DATA

	COLLECTION SOURCE			
DATA FIELD	SAMTRAC[1]	TR[2] LC	LAB[3] TAPE	GENERATED
Amount			X	
Amount (Library)			X	
Bottle Number			X	
Carrier Gas Flow Rate			X	
Column Final Hold			X	
Column Initial Hold			X	
Column Inside Diameter			X	
Column Length			X	
Column Temperature Program			X	
Compound Comment Code		X		
Compound Name				X
Compound Number			X	
Compound Reference Number			X	
Compound Type				X
Concentration/Dilution Factor			X	
Date Analyzed			X	
Date Extracted			X	
Date Sampled		X		
Episode Comment Codes		X		
Episode Number	X			
Fraction			X	
Fraction Comment Code		X		
Industrial Category Code	X			
Instrument			X	
Laboratory	X			

---

(1) SAMTRAC - Computerized logistics system at the Sample Control Center.

(2) TR LC - Traffic Reports and Lab Chronicles.

(3) Quantitation Reports on magnetic tape received from analytical laboratories.

SUMMARY OF DATA SOURCES FOR COLLECTION OF ORGANIC PRIORITY POLLUTANT DATA

	COLLECTION SOURCE			
DATA FIELD	SAMTRAC[1]	TR[2] LC	LAB[3] TAPE	GENERATED
Mass to Charge Ratio			X	
Method			X	
Peak Area			X	
PH Level		X		
Plant Code	X			
Proprietary Indicator		X		
Quantitation Report Type			X	
Reference Compound			X	
Relative Retention Time			X	
Relative Retention Time (Library)			X	
Response Factor			X	
Response Factor (Library)			X	
Retention Time			X	
Retention Time (Library)			X	
Sample Comment Code		X		
Sample Number	X	X	X	
Sample Point (Site)		X		
Sample Point Flow		X		
Sample Type			X	
Scan Number			X	
Shift			X	
Time Analyzed			X	
Unit of Measure			X	

---

(1) SAMTRAC - Computerized logistics system at the Sample Control Center.

(2) TR LC - Traffic Reports and Lab Chronicles.

(3) Quantitation Reports on magnetic tape received from analytical laboratories.

NOTES ON TYPE/LENGTH DESCRIPTION

The Type/Length Description for each EGD Data Element represents how each data field is stored internally in the computer or how each data field is represented on the quantitation report.

TYPES:

Z - Numeric data only - leading zeroes not printed.
9 - Numeric data only - zeroes printed.
X - Alpha numeric data.
V - Implied decimal point.
. - Explicit decimal point.

LENGTH:

(N) Where N is a positive integer value from 1 to 255, gives the number of data positions allocated internally by the computer to store this portion of the data field.

EXAMPLES:

Example 1. 9(7)V9(3)

9 - Numeric data.
(7)+(3) - 10 data positions allocated.
V - Implied decimal point after 3rd position from the right.

Can also be expressed as 9999999V999.

The number 1,130.31 would be stored internally under this Type/Length description as:

'0001130310'

NOTES ON TYPE/LENGTH DESCRIPTION (CONT.)

The computer program would also know that there is a decimal point implied between '0001130' and '310'.

Example 2. X(6)

X - Alphanumeric data.
(6) - 6 data positions allocated.

Can also be expressed as XXXX.

The field 'EPA1' would be stored internally or printed as:

'EPA1'

Example 3. ZZZZZZ9.999

Z - Numeric data - zeroes not printed.
9 - Numeric data - zeroes printed.
. - Explicit decimial point printed.
6 (Z's) + 4 (9's) + 1 (.) = 11 data positions allowed.

The field '0000023010' would be printed as:

'   23.010'

ELEMENT NAME: AMOUNT

Definition: The quantitative measurement of the compound determined by GC or GC/MS analysis. The amount is computed for the compound using the referenced internal standard or isotopic diluent and is multiplied by the concentration or dilution factor to yield final solution concentration in ug/L.

Input	**Type/Length**
Quantitation Report	ZZZZZ9.999
As Stored Internally	9(7)V9(3)

Unit of Measure

Ug/L

Edit Criteria:

Range: 10.000-999,999.999 ug/L

Examples:

Volatiles: Concentration (AMOUNT) is reported on the quantitation report in uG/L. If the sample is diluted to bring a pollutant within the analytical range of the instrument, the AMOUNT is multiplied by the concentration/dilution factor (see CONCENTRATION/DILUTION FACTOR).

Semi-volatiles: Concentration (AMOUNT) is reported on the quantitation report in uG/ML. If the sample or extract is diluted to bring a pollutant within the analytical range of the instrument, the AMOUNT is multiplied by the concentration/dilution factor (see CONCENTRATION/DILUTION FACTOR).

ELEMENT NAME: AMOUNT (LIBRARY)

Definition: The reference amount in the standard and the amount on which quantitation is based.

Input	Type/Length
Quantitation Report	ZZZZZ9.99
As Stored Internally	9(7)V9(3)

Unit of Measure

The amount is reported as a pure number but must always be accompanied by a UNIT. See UNIT.

Edit Criteria:

Range: 1.000 - 1000.000 ug/L

Examples: See AMOUNT.

ELEMENT NAME: BOTTLE NUMBER

Definition: A numeric code which uniquely identifies the bottles used for a particular sample. Used as a suffix to the SAMPLE NUMBER.

Input	**Type/Length**
Quantitation Report	X(2)
As Stored Internally	X(2)

Unit of Measure

Each.

Edit Criteria:

Range: 01-98 for EPA Samples
99 for the TCLP Leachate of an EPA Sample
00 for all Others

Example: See SAMPLE NUMBER.

ELEMENT NAME: CARRIER GAS FLOW RATE

Definition: The volumetric (volume/time) rate of flow of the carrier gas in the gas chromatograph for packed columns, or the linear gas velocity (distance/time) for capillary columns.

Input	**Type/Length**
Quantitation Report	X(9)
As Stored Internally	X(9)

Unit of Measure

Volatiles (packed column): ML/Min
Semi-volatiles (capillary column): CM/Sec

Edit Criteria:

Ranges: Volatiles: 20-40 mL/min; Semi Volatiles: 20-60 cm/sec; Dioxin: 20-60 cm/sec

ELEMENT NAME: COLUMN FINAL HOLD

Definition: The final temperature of the gas chromatograph column and the length of time that it was held.

Input	**Type/Length**
Quantitation Report	X(7)
As Stored Internally	X(7)

Unit of Measure

Time: minutes
Temperature: degrees Celsius
Units are understood and not reported.

Edit Criteria:

1. Format: Hold @ temperature ie XXX@XXX

2. Hold Ranges: Volatiles: 0-20 min; Semi Volatiles: 10-20 min; Dioxin: 10-30 min

3. Temperature Ranges: Volatiles: 200-250°C; Semi Volatiles: 240-290°C; Dioxin: 200-300°C

Example: 15 @ 280 means that the column was held for 15 minutes.

ELEMENT NAME: COLUMN INITIAL HOLD

Definition: The initial temperature of the gas chromatograph column and the length of time that it was held.

Input	**Type/Length**
Quantitation Report	X(7)
As Stored Internally	X(7)

Unit of Measure

Time: minutes
Temperature: degrees Celsius
Units are understood and not reported.

Edit Criteria:

1. Format: Hold @ Temp ie XXX@XXX

2. Hold Ranges: Volatiles: 2-4 min; Semi Volatiles: 4-6 min; Dioxin: 0-10 min

3. Temperature Ranges: Volatiles: 25-50°C; Semi Volatiles: 25-35°C; Dioxin: 50-200°C

Example: 4 @ 30 means that the column was held for 4 minutes at 30°C.

ELEMENT NAME: COLUMN INSIDE DIAMETER

Definition: The internal diameter of the gas chromatograph column.

Input	**Type/Length**
Quantitation Report	X(6)
As Stored Internally	X(6)

Unit of Measure

Millimeter (MM)

Edit Criteria:

Ranges: Volatiles: 1-3mm; Semi-Volatiles: 0.2-0.35 mm; Dioxin: 0.2-0.35 mm

ELEMENT NAME: COLUMN LENGTH

Definition: The length of the gas chromatograph column.

Input	**Type/Length**
Quantitation Report	X(6)
As Stored Internally	X(6)

Unit of Measure

Meters (M)

Edit Criteria:

Ranges: Volatiles: 2.8-3.1 m; Semi-Volatiles: 25-35 m; Dioxin: 25-65 m

ELEMENT NAME: COLUMN TEMPERATURE PROGRAM

Definition: The change in column temperature with respect to time giving the initial and final column temperatures.

Input	**Type/Length**
Quantitation Report	X(10)
As Stored Internally	X(10)

Unit of Measure

Initial temperature: degrees Celsius
Final temperature: degrees Celsius
Rate: degrees Celsius per minute
Units are understood and not reported.

Edit Criteria:

1. Format: Initial Temp - Final Temp @ Temp Program rate ie XXX-XXX @ XX

2. Range: 1.5-8.5°C/min

Examples:

45-220 @ 8
30-280 @ 8

ELEMENT NAME: COMPOUND COMMENT CODE

Definition: A coded value for any optional text that may be associated with each compound.

Input	**Type/Length**
Traffic Report	X(4)
Laboratory Chronicles	
As Stored Internally	X(4)

Unit of Measure

N/A

Edit Criteria:

Range C001-C999.

ELEMENT NAME: COMPOUND NAME

Definition: The name of the compound determined. The compound name corresponds to the EGD compound number.

Input	**Type/Length**
Quantitation Report	X(30)
As Stored Internally	X(30)

Unit of Measure

N/A

Edit Criteria:

See ITD Compound Code Table, attached as Table 4, for valid names.

Examples:

BROMOFORM
1,2-DICHLORBENZENE-D4

ELEMENT NAME: COMPOUND NUMBER

Definition: A numerical code which uniquely identifies each unique chemical compound.

Input

	Type/Length
Quantitation Report	9(3)
As Stored Internally	9(3)

Unit of Measure

N/A

Edit Criteria:

Must be one of the following codes:

001-129 = Priority Pollutants quantitated by internal standard.
130-199 = Miscellaneous surrogates and internal standards.
200-299 = Priority Pollutant labeled compounds (isotopes) quantitated by internal standard.
300-399 = Priority Pollutants quantitated by isotope dilution.
400-429 = Labeled compounds (isotopes) quantitated by internal standard.
500-599 = Non-Priority Pollutant compounds quantitated by internal standard.
600-699 = Syn Fuel specific and Appendix C labeled compounds (isotopes) quantitated by internal standard.
700-799 = Syn Fuel specific and Appendix C compounds quantitated by isotope dilution.
800-829 = Pollutants 100-129 quantitated by isotope dilution.
900-999 = Non-Priority Pollutant Compounds Quantitated by Internal standard.

See ITD Compound Code Table, attached as Table 4.

ELEMENT NAME: COMPOUND ORDER NUMBER

Definition: A numerical code that establishes the order of compound determination by the GC/MS. The code is used on the Quantitation Report to match the segments of compound data within the report.

Input	**Type/Length**
Quantitation Report	9(3)
As Stored Internally	9(3)

Unit of Measure

N/A

Edit Criteria:

Range: 001-250

ELEMENT NAME: COMPOUND TYPE

Definition: A coded value which identifies a chemical compound as a priority pollutant or surrogate.

Input	**Type/Length**
Generated Based on Compound Number	X(1)
As Stored Internally	X(1)

Unit of Measure

N/A

Edit Criteria:

Must be one of the following codes:

CODE	VALUE
D	Isotopic Diluent
I	Internal Standard
P	Priority Pollutant
S	Surrogate

ELEMENT NAME: CONCENTRATION/DILUTION FACTOR

Definition: The concentration or dilution ratio of the sample fraction or spike before analysis.

Input	**Type/Length**
Quantitation Report	X(11)
As Stored Internally	9(5)V9(5)

Unit of Measure

Pure number.

Edit Criteria:

Format: two numbers separated by a colon, the first is the pre-extraction/dilution amount; the second is the post extraction/dilution amount, e.g., XXXXX:XXXXX. These numbers are used to form the concentration/dilution ratio (CDR); the number to the left of the colon becomes the numerator, and the number to the right of the colon becomes the denominator.

Examples: 1000:1 means that an initial 1000mL volume of sample was concentrated to 1 mL extract (semi-volatiles); 1:100 means that an inital volume of sample was diluted with 99 parts of reagent water (volatiles).

Semi-Volatiles: When a semi-volatiles fraction (acid or base/neutral) is extracted, the normal volume of sample is 1 liter (1000 mL). The extract is normally concentrated to a volume of 1.0 mL. The units on the quantitiation report for semi-volatiles is UG/ML. Multiplying these units by 1000 mL/L, and dividing the concentration/dilution ration (CDR) (1000/1) yields the final concentration in the water in ug/L, as given in the following equation:

Csam (ug/L) = Cext (ug/mL) x 1000 (mL/L) / CDR

ELEMENT NAME: CONCENTRATION/DILUTION FACTOR (Continued)

Edit Criteria (continued):

For example, for a 1.0 mL extract containing 67 ug/mL of a pollutant which was extracted from a 1000 mL water sample:

Csam (ug/mL) = 67 ug/mL x 1000 mL/L / 1000 = 67 ug/L

When a sample volume other than 1000 mL is extracted, the volume is given on the left hand side of the colon in the concentration/dilution factor. For example, for the "dilute aliquot" in Method 1625A which employs a 10 mL volume of sample and yields 1 mL of extract, the concentration/dilution factor would be: 10:1, and the CDR would be 10.

Similarly, when an extract is diluted, the dilute volume is given on the right hand side of the colon in the concentration/dilution factor. For example, for a 1 1000 mL sample which yields a final extract volume of 1.0 mL which is then diluted by a factor of 10, the concentration/dilution factor would be 1000:10, and the CDR would become 1000/10, or 100. Extract volumes other than multiples of 10 of the 1.0 mL extract volume are prohibited.

For semi-volatiles, the 2,2'-difluorobiphenyl internal standard concentration remains constant at 100 ug/mL in the extract, regardless of the level of dilution.

Computations of pollutant and labeled compound concentrations become complicated when the initial volume of sample is other than 1000 mL or 1000 divided by some multiple of 10, and even more complicated when the final extract volume is not exactly 1.0, but is close to 1.0 mL (e.g., in the range of 0.5 to 2.0 mL). These situations are dealt with as follows:

1. When the sample volume is not 1000 mL, but is adjusted to 1000 mL prior to spike of the labeled compounds and extraction, the concentration for each pollutant will be (see note below!) adjusted by the CDR, but the final concentration for the labeled compounds will not be adjusted.

ELEMENT NAME: CONCENTRATION/DILUTION FACTOR (Continued)

Edit Criteria (continued):

2. When the final extract volume is not exactly 1.0 mL, the concentration for each pollutant determined by isotope dilution will not be (see note below!) adjusted by the right hand portion of the concentration/dilution factor (since the labeled compound compensates the pollutant recovery), but the concentration (and therefore the recovery) of each labeled compound and each pollutant with no labeled analog will be adjusted by the right hand portion of the concentration/dilution factor. Extract volumes of less than 0.5 and in the range of 2.0 to 9.9 mL are prohibited.

NOTE: The adjustments referenced above will be made by the EPA computer after data are received. In order to minimize the workload for the laboratories, no adjustments to concentrations are to be made by the laboratories. However, it is imperative that the 2.2'-difluorobiphenyl concentration be 100 ug/mL in the extract (as is required by Method 1625), that the compound numbers be correct (so that the quantitation method, i.e., isotope dilution or internal standard can be identified), and that the initial volume of sample and final volume of extract be stated correctly in the concentration/dilution factor, so that the final concentration in the water is computed properly using the information above. If you are confused by all of this, the simplest way to avoid these issues is to always use 1000 mL of sample, always concentrate to 1.0 mL, and always spike the 2,2'-difluorobiphenyl such that its concentration in the extract will be 100 ug/mL.

Finally, the amount of each labeled compound in the water which is extracted shall be 100 ug only. Other amounts are prohibited. This requirement recognizes that the "sample" may not always be 1000 mL (e.g., the dilute aliquot), but that the water in the extractor will yield an extract with concentrations of the labeled compounds identical to those in the precision and recovery standards.

Volatiles: For volatiles, the labeled compounds and internal standards are spiked into the 5 mL water sample at each level of dilution. Therefore, the computation of concentration is applied to the pollutants only (not to the labeled compounds or internal standards).

ELEMENT NAME: CONCENTRATION/DILUTION FACTOR (Continued)

Edit Criteria (continued):

In a way analogous to that for semi-volatiles, the final concentration of a pollutant in a sample can be expressed as follows:

Csam (ug/L) = Cdil samp (ug/L) / CDR

For example, for a sample which has been diluted by a factor of 10 with reagent water and contains 48 ug/L at the dilute level, the concentration/dilution factor is 1:10 and the CDR is 1/10 or 0.1.

Csam (ug/L) = 48 ug/L / 0.1 = 480 ug/L

NOTE: The "concentration/dilution factor" format was developed to permit use of the same "units" on quantitation reports, regardless of how much the sample is concentrated or diluted. Alternate methods require changing the units depending on the amount of the dilution.

ELEMENT NAME: DATE ANALYZED

Definition: The date that the sample fraction was analyzed by the laboratory.

Input	**Type/Length**
Quantitation Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: MM/DD/YY, where MM is the month; DD is the day; and YY is the last two digits of the Gregorian calendar year.

Example: 07/15/83 is July 15, 1983.

ELEMENT NAME: DATE EXTRACTED

Definition: The date that the laboratory extracted the sample for analysis.

Input	**Type/Length**
Quantitation Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: MM/DD/YY, where MM is the month; DD is the day; and YY is the last two digits of the Gregorian calendar year.

Example: 07/15/83 is July 15, 1983.

ELEMENT NAME: DATE SAMPLED

Definition: The date the sample was taken by the field sampler.

Input	**Type/Length**
Traffic Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: MM/DD/YY, where MM is the month; DD is the day; and YY is the last two digits of the Gregorian calendar year.

Example: 07/15/83 is July 15, 1983.

ELEMENT NAME: EPISODE COMMENT CODE

Definition: A coded value for comments associated with an episode.

Input	Type/Length
Traffic Reports	X(4)
Laboratory Chronicles	
As Stored Internally	X(4)

Unit of Measure

N/A

Edit Criteria:

Range E001-E999.

ELEMENT NAME: EPISODE NUMBER

Definition: The SCC assigned identification code designating the sampling trip.

Input	**Type/Length**
SAMTRAC	9(4)
As Stored Internally	9(4)

Unit of Measure

N/A

Edit Criteria:

1. Numeric
2. Greater than 0119.

ELEMENT NAME: FRACTION

Definition: A coded value which designates the compound as either an acid, base/neutral, volatile, pesticide or dioxin.

Input	**Type/Length**
Priority Pollutant Data Sheet	X(1)
QA/QC Sheet	
As Stored Internally	X(1)

Unit of Measure

N/A

Edit Criteria:

Must be one of the following codes:

CODE	VALUE
A	Acid Compound
B	Base/Neutral Compound
C	Combined Acid Base/Neutral
D	Dioxin
P	Pesticide Compound
V	Volatile Compound

ELEMENT NAME: FRACTION COMMENT CODE

Definition: A coded value for any optional text that may be associated with each fraction.

Input	Type/Length
Traffic Report/Lab Chronicles	X(4)
Priority Pollutant Data Sheet	
As Stored Internally	X(4)

Unit of Measure

N/A

Edit Criteria:

Range F001-F999.

ELEMENT NAME: INDUSTRIAL CATEGORY CODE

Definition: The classification of the industrial processes performed by the plant where a sample was taken.

Input	**Type/Length**
SAMTRAC	9(3)
As Stored Internally	9(3)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the Industrial Category Code Table.

ELEMENT NAME: INSTRUMENT

Definition: A coded value assigned by the laboratory that uniquely identifies each GC/MS instrument within a laboratory. All Calibration, Precision and Recovery, Standards and Blank Quantitation files will be tracked by this instrument member within Laboratory. Changing of this instrument number by the laboratory would necessitate the submittal of new calibration and other initial quantitation runs by the laboratory.

Input	**Type/Length**
Quantitation Report	X(2)
As Stored Internally	X(2)

Unit of Measure

N/A

Edit Criteria:

Range: 01-99, AA-ZZ or any two character combination.

ELEMENT NAME: LABORATORY

Definition: A numerical code used to identify the specific laboratory where the sample was analyzed.

Input	**Type/Length**
SAMTRAC	9(3)
As Stored Internally	9(3)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the Laboratory Code Table.

ELEMENT NAME: MASS TO CHARGE RATIO

Definition: Designates the quantitation ion. Abbreviated as M/Z, or M/E.

Input	**Type/Length**
Quantitation Report	ZZZ9
As Stored Internally	9(4)

Unit of Measure

N/A

Edit Criteria:

Ranges: Volatiles: 20-250; Semi-Volatiles: 35-450; Dioxin (Selected Ion Monitoring): 257, 320, 322, 323, 328, 332, and 334.

ELEMENT NAME: METHOD

Definition: A coded value which uniquely identifies the method protocol that was followed during analysis.

Input	**Type/Length**
Quantitation Report	X(5)
As Stored Internally	X(5)

Unit of Measure

N/A

Edit Criteria:

Acceptable Codes:

1624A
1625A
1624B
1625B
1624C
1625C
1634
1635
613
613E
713

ELEMENT NAME: PEAK AREA

Definition: The area beneath the peak of a mass chromatogram. The peak area is proportional to the amount of the detected compound at an observed mass to charge ratio. It is used to compute the concentration of the compound present in the sample.

Input	**Type/Length**
Quantitation Report	ZZZZZZZZZZ9.
As Stored Internally	9(10)

Unit of Measure

N/A

Edit Criteria:

ELEMENT NAME: PH LEVEL

Definition: The negative logarithm of the effective hydrogen ion concentration as expressed in grain equivalents per liter.

Input	**Type/Length**
Traffic Report	Z9.999
As Stored Internally	9(2)V9(3)

Unit of Measure

N/A

Edit Criteria:

ELEMENT NAME: PLANT CODE

Definition: A numeric code used to distinguish specific industrial plants which have been sampled.

Input	**Type/Length**
SAMTRAC	9(4)
As Stored Internally	9(4)

Unit of Measure

N/A

Edit Criteria:

Must be a four digit number.

Comment:

1. Plant ID's are unique within the isotope dilution program.

2. The episode number for the first occurrence of a plant visit is used as the plant's identification number.

ELEMENT NAME: PROPRIETARY INDICATOR

Definition: A coded value which designates whether or not the analysis data from a sample is proprietary. Also indicates that confidentiality papers have been signed.

Input	**Type/Length**
Traffic Report	X(1)
Lab Chronicles	
As Stored Internally	X(1)

Unit of Measure

N/A

Edit Criteria:

Must be one of the following codes:

CODE	VALUE
P	Proprietary
N	Not Proprietary

ELEMENT NAME: QUANTITATION REPORT TYPE

Definition: A coded value that uniquely identifies the particular type of quantitation report that is being submitted.

Input	**Type/Length**
Quantitation Report	X(3)
As Stored Internally	X(3)

Unit of Measure

N/A

Edit Criteria:

CODE	VALUE
BLK	Laboratory Blank
CAL	Calibration
EPA	EPA Sample
FBK	Field Blank
VER	Calibration Verification for Extraction Methods
IPR	Initial Precision and Accuracy
OPR[1]	Ongoing Precision and Accuracy

[1] For volatiles, calibration verification and ongoing precision and accuracy tests are performed using the OPR report.

ELEMENT NAME: REFERENCE COMPOUND

Definition: A numeric code that is used as a pointer to the internal standard or isotopic diluent within a quantitation report.

Input	**Type/Length**
Quantitation Report	ZZ9
As Stored Internally	9(3)

Unit of Measure

N/A

Edit Criteria:

Range: 001-250

ELEMENT NAME: RELATIVE RETENTION TIME

Definition: The quotient of the retention time of a compound divided by its internal standard or isotopic diluent.

Input	**Type/Length**
Quantitation Report	Z9.999
As Stored Internally	99V9(3)

Unit of Measure

N/A

Edit Criteria:

ELEMENT NAME: RELATIVE RETENTION TIME (LIBRARY)

Definition: The relative retention time stored in the library. The value is based on the analysis of a standard containing both compounds.

Input	**Type/Length**
Quantitation Report	Z9.999
As Stored Internally	9(2)V9(3)

Unit of Measure

N/A

Edit Criteria:

ELEMENT NAME: RESPONSE FACTOR

Definition: The ratio between the response for the sample and a response for a standard under identical analytical conditions. Computed per the following equation:

$$RF = \frac{A_S C_{IS}}{A_{IS} C_S}$$

where

A_S is the PEAK AREA for the compound from analysis of a standard.
A_{IS} is the PEAK AREA for the internal standard.
C_{IS} is the concentration of the internal standard.
C_S is the concentration of the compound.

Input	**Type/Length**
Quantitation Report	ZZZ9.99
As Stored Internally	9(4)V9(3)

Unit of Measure

N/A

Edit Criteria:

ELEMENT NAME: RESPONSE FACTOR (LIBRARY)

Definition: The response factor stored in the library. The value is determined from analysis of a standard.

Input	**Type/Length**
Quantitation Report	ZZZ9.999
As Stored Internally	9(4)V9(3)

Unit of Measure

N/A

Edit Criteria:

Example: See RESPONSE FACTOR.

ELEMENT NAME: RETENTION TIME

Definition: The time it takes the identified compound to elute from the gas chromatograph.

Input	**Type/Length**
Quantitation Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: HH:MM:SS
MM:SS
SS

Where HH is hours; MM is minutes; SS is seconds.

ELEMENT NAME: RETENTION TIME (LIBRARY)

Definition: The known time it takes an identified compound to elute from the gas chromatograph. The time is determined from analysis of a standard.

Input	**Type/Length**
Quantitation Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: HH:MM:SS
MM:SS
SS

Where HH is hours; MM is minutes; SS is seconds.

ELEMENT NAME: SAMPLE COMMENT CODE

Definition: A coded value for any optional text that may be associated with each sample.

Input	**Type/Length**
Traffic Reports Lab Chronicles	X(4)
As Stored Internally	X(4)

Unit of Measure

N/A

Edit Criteria:

Range S001-S999.

ELEMENT NAME: SAMPLE NUMBER

Definition: The SCC assigned identification code which identifies the individual samples. For calibration and performance standards, is used to indicate the nominal concentration of the standard.

Input	**Type/Length**
Quantitation Report	ZZ999
As Stored Internally	9(5)

Unit of Measure

N/A

Edit Criteria:

a. Must be a five digit number.

b. Range: 00001-99999

Examples: 00100 accompanied by a QUANTITATION REPORT TYPE of VER would define a Calibration Verification standard at a nominal concentration of 100 ug/mL (or 100 ug/L for volatiles).

ELEMENT NAME: SAMPLE POINT FLOW

Definition: The flow rate at the point at which the sample was taken. Value is recorded from a flow meter or other flow measuring device.

Input	**Type/Length**
Traffic Report	X(5)
As Stored Internally	X(5)

Unit of Measure

Per 1,000 gallons/day.

Edit Criteria:

ELEMENT NAME: SAMPLE POINT (SITE)

Definition: The specific point within an industrial wastestream where a sample was taken.

Input	**Type/Length**
Traffic Report	X(1)
Lab Chronicles	
As Stored Internally	X(1)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the Sample Site Table.

ELEMENT NAME: SAMPLE TYPE

Definition: A coded value which describes the type of sample.

Input	**Type/Length**
Traffic Report	X(2)
As Stored Internally	X(2)

Unit of Measure

N/A

Edit Criteria:

Must be a valid code in the Sample Type Code Table.

ELEMENT NAME: SCAN NUMBER

Definition: Gives the scan at which the compound was detected by the mass spectrometer.

Input	**Type/Length**
Quantitation Report	ZZ999
As Stored Internally	9(5)

Unit of Measure

N/A

Edit Criteria:

Range: 00001-99999

ELEMENT NAME: SHIFT

Definition: The scheduled period of operation of the GC/MS instrument. Operation is divided into three shifts/day.

Input	**Type/Length**
Quantitation Report	X(1)
As Stored Internally	X(1)

Unit of Measure

N/A

Edit Criteria:

Code	Meaning
G	Graveyard (0000-0759; Midnight to 8 AM)
D	Day (0800 - 1559; 8 AM to 4 PM)
S	Swing (1600 - 1159; 4 PM to Midnight)

ELEMENT NAME: TIME ANALYZED

Definition: The time thtat the sample fraction was analyzed by the laboratory.

Input	**Type/Length**
Quantitation Report	X(8)
As Stored Internally	X(8)

Unit of Measure

N/A

Edit Criteria:

Format: HH:MM:SS

ELEMENT NAME: UNIT OF MEASURE

Definition: The unit of measurement for the amount, as measured in the nominal sample.

Input	**Type/Length**
Quantitation Report	X(5)
As Stored Internally	X(5)

Unit of Measure

Volatiles: UG/L
Semi Volatiles: UG/ml
Dioxin: NG/ML

Edit Criteria:

UG/L
UG/ML
NG/ML

COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	001	B	ACENAPHTHENE	P	0154	Y
04	002	V	ACROLEIN	P	0056	Y
04	003	V	ACRYLONITRILE	P	0053	Y
04	004	V	BENZENE	P	0078	Y
04	005	B	BENZIDINE	P	0184	Y
04	006	V	CARBON TETRACHLORIDE	P	0047	Y
04	007	V	CHLOROBENZENE	P	0112	Y
04	008	B	1,2,4-TRICHLOROBENZENE	P	0180	Y
04	009	B	HEXACHLOROBENZENE	P	0284	Y
04	010	V	1,2-DICHLOROETHANE	P	0062	Y
04	011	V	1,1,1-TRICHLOROETHANE	P	0097	Y
04	012	B	HEXACHLOROETHANE	P	0201	Y
04	013	V	1,1-DICHLOROETHANE	P	0063	Y
04	014	V	1,1,2-TRICHLOROETHANE	P	0083	Y
04	015	V	1,1,2,2-TETRACHLOROETHANE	P	0083	Y
04	016	V	CHLOROETHANE	P	0064	Y
04	017	V	BIS (CHLOROMETHYL) ETHER (NR)	P		Y
04	018	B	BIS(2-CHLOROETHYL)ETHER	P	0093	Y
04	019	V	2-CHLOROETHYLVINYL ETHER	P	0106	Y
04	020	B	2-CHLORONAPHTHALENE	P	0162	Y
04	021	A	2,4,6-TRICHLOROPHENOL	P	0196	Y
04	022	A	4-CHLORO-3-METHYLPHENOL	P	0107	Y
04	023	V	CHLOROFORM	P	0085	Y
04	024	A	2-CHLOROPHENOL	P	0128	Y
04	025	B	1,2-DICHLOROBENZENE	P	0146	Y
04	026	B	1,3-DICHLOROBENZENE	P	0146	Y
04	027	B	1,4-DICHLOROBENZENE	P	0146	Y
04	028	B	3,3'-DICHLOROBENZIDINE	P	0252	Y
04	029	V	1,1-DICHLOROETHENE	P	0061	Y
04	030	V	TRANS-1,2-DICHLOROETHENE	P	0061	Y
04	031	A	2,4-DICHLOROPHENOL	P	0162	Y
04	032	V	1,2-DICHLOROPROPANE	P	0063	Y
04	033	V	T-1,3-DICHLOROPROPENE	P	0075	Y
04	034	B	2,4-DIMETHYLPHENOL	P	0122	Y
04	035	B	2,4-DINITROTOLUENE	P	0165	Y
04	036	B	2,6-DINITROTOLUENE	P	0165	Y
04	037	B	1,2-DIPHENYLHYDRAZINE	P	0077	Y
04	038	V	ETHYLBENZENE	P	0106	Y
04	039	B	FLUORANTHENE	P	0202	Y
04	040	B	4-CHLOROPHENYL PHENYL ETHER	P	0204	Y
04	041	B	4-BROMOPHENYL PHENYL ETHER	P	0248	Y
04	042	B	BIS (2-CHLOROISOPROPYL) ETHER	P	0121	Y
04	043	B	BIS (2-CHLOROETHOXY) METHANE	P	0093	Y
04	044	V	METHYLENE CHLORIDE	P	0084	Y
04	045	V	CHLOROMETHANE	P	0050	Y
04	046	V	BROMOMETHANE	P	0096	Y
04	047	V	BROMOFORM	P	0173	Y
04	048	V	BROMODICHLOROMETHANE	P	0083	Y
04	049	V	TRICHLOROFLUOROMETHANE (NR)	P		Y
04	050	V	DICHLORODIFLUOROMETHANE (NR)	P		Y

COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	051	V	DIBROMOCHLOROMETHANE	P	0129	Y
04	052	B	HEXACHLORO-1,3-BUTADIENE	P	0225	Y
04	053	B	HEXACHLOROCYCLOPENTADIENE	P	0237	Y
04	054	B	ISOPHORONE	P	0082	Y
04	055	B	NAPHTHALENE	P	0128	Y
04	056	B	NITROBENZENE	P	0123	Y
04	057	A	2-NITROPHENOL	P	0139	Y
04	058	A	4-NITROPHENOL	P	0139	Y
04	059	A	2,4-DINITROPHENOL	P	0184	Y
04	060	A	2-METHYL-4,6-DINITROPHENOL	P	0198	Y
04	061	B	N-NITROSODIMETHYLAMINE	P	0074	Y
04	062	B	N-NITROSODIPHENYLAMINE	P	0169	Y
04	063	B	N-NITROSODI-N-PROPYLAMINE	P	0070	Y
04	064	A	PENTACHLOROPHENOL	P	0266	Y
04	065	B	PHENOL	P	0094	Y
04	066	B	BIS (2-ETHYLHEXYL) PHTHALATE	P	0149	Y
04	067	B	BUTYL BENZYL PHTHALATE	P	0149	Y
04	068	B	DI-N-BUTYL PHTHALATE	P	0149	Y
04	069	B	DI-N-OCTYL PHTHALATE	P	0149	Y
04	070	B	DIETHYL PHTHALATE	P	0149	Y
04	071	B	DIMETHYL PHTHALATE	P	0163	Y
04	072	B	BENZO(A)ANTHRACENE	P	0228	Y
04	073	B	BENZO(A)PYRENE	P	0252	Y
04	074	B	BENZO(B)FLUORANTHENE	P	0252	Y
04	075	B	BENZO(K)FLUORANTHENE	P	0252	Y
04	076	B	CHRYSENE	P	0228	Y
04	077	B	ACENAPHTHYLENE	P	0152	Y
04	078	B	ANTHRACENE	P	0178	Y
04	079	B	BENZO(GHI)PERYLENE	P	0276	Y
04	080	B	FLUORENE	P	0166	Y
04	081	B	PHENANTHRENE	P	0178	Y
04	082	B	DIBENZO(A,H)ANTHRACENE	P	0278	Y
04	083	B	INDENO(1,2,3-CD)PYRENE	P	0276	Y
04	084	B	PYRENE	P	0202	Y
04	085	V	TETRACHLOROETHENE	P	0164	Y
04	086	V	TOLUENE	P	0092	Y
04	087	V	TRICHLOROETHENE	P	0095	Y
04	088	V	VINYL CHLORIDE	P	0062	Y
04	089	P	ALDRIN	P		Y
04	090	P	DIELDRIN	P		Y
04	091	P	CHLORDANE	P		Y
04	092	P	4,4'-DDT	P		Y
04	093	P	4,4'-DDE	P		Y
04	094	P	4,4'-DDD	P		Y
04	095	P	ALPHA-ENDOSULFAN	P		Y
04	096	P	BETA-ENDOSULFAN	P		Y
04	097	P	ENDOSULFAN SULFATE	P		Y
04	098	P	ENDRIN	P		Y
04	099	P	ENDRIN ALDEHYDE	P		Y
04	100	P	HEPTACHLOR	P		Y

COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	101	P	HEPTACHLOR EPOXIDE	P		Y
04	102	P	ALPHA-BHC	P		Y
04	103	P	BETA-BHC	P		Y
04	104	P	GAMMA-BHC	P		Y
04	105	P	DELTA-BHC	P		Y
04	106	P	PCB-1242	P		Y
04	107	P	PCB-1254	P		Y
04	108	P	PCB-1221	P		Y
04	109	P	PCB-1232	P		Y
04	110	P	PCB-1248	P		Y
04	111	P	PCB-1260	P		Y
04	112	P	PCB-1016	P		Y
04	113	P	TOXAPHENE	P		Y
04	129	D	2,3,7,8-TCDD	P	0320	Y
04	130	V	XYLENES	P		Y
04	150	A	PHENOL-D5	S		Y
04	151	A	PENTAFLUOROPHENOL	S		Y
04	152	V	PENTAFLOUROBENZENE	S		Y
04	153	A	TRIFLUORO-M-CRESOL	S		Y
04	154	V	2,2-DIFLUOROTETRACHLOROETHANE	S		Y
04	155	B	2-FLUOROBIPHENYL	S		Y
04	156	B	1-FLUORONAPHTHALENE	S		Y
04	157	A	2-FLOUROPHENOL	S		Y
04	158	B	2-FLUORONAPHTHALENE	S		Y
04	159	B	PYRIDINE-D5	S		Y
04	160	B	ANILINE-D5	S		Y
04	161	B	NAPHTHALENE-D8	S		Y
04	162	V	TOLUENE-D8	S		Y
04	163	B	NITROBENZENE-D5	S		Y
04	164	B	2,2'-DIFLUOROBIPHENYL	I	0190	Y
04	165	V	BENZENE-D6	S		Y
04	166	B	DECAFLUOROBIPHENYL	S		Y
04	167	V	M-DIFLUOROBENZENE	S		Y
04	163	V	METHYLENE CHLORIDE-D2	S		Y
04	169	V	1,1,2,2-TETRACHLOROETHANE-D2	S		Y
04	170	V	ETHYLBENZENE-D10	S		Y
04	172	V	1,2 DICHLOROETHANE-D4	S		Y
04	173	V	2,2 DICHLOPROPANE-D6	S		Y
04	174	V	CHLOROBENZENE-D5	S		Y
04	175	B	1,2 DICHLOROBENZENE-D4	S		Y
04	176	B	CHRYSENE D12	S		Y
04	177	B	FLUORENE D10	S		Y
04	178	A	2-NITROPHENOL D4	S		Y
04	179	B	DI-N-BUTYL-PHTHALATE-D4	S		Y
04	180	B	4-FLUOROANILINE	S		Y
04	181	V	BROMOCHLOROMETHANE	I	0128	Y
04	182	V	2-BROMO-1-CHLOROPROPANE	I	0077	Y
04	183	V	1,4-DICHLOROBUTANE	I	0055	Y
04	184	D	2,3,7,8-TCDD-37CL4	I	0328	Y
04	185	V	BROMOFLUOROBENZENE	I	0095	Y

10/30/86

ISOTOPE DILUTION

COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	186	B	DFTPP	I	0198	N
04	187	P	2'-CHLOROBIPHENYL-D5	S		N
04	188	P	3',4',5'-TRICHLOROBIPHENYL-D5	S		N
04	189	P	3,3',4,4'-TETRACHLOROBIPHEN-D6	S		N
04	201	B	ACENAPHTHENE-D10	D	0164	Y
04	202	V	ACROLEIN-D4	D	0060	Y
04	203	V	ACRYLONITRILE-D3	D	0056	Y
04	204	V	BENZENE-D6	D	0084	Y
04	205	B	BENZIDINE (RINGS-D8)	D	0192	Y
04	206	V	CARBON TETRACHLORIDE-13C	D	0048	Y
04	207	V	CHLOROBENZENE-D5	D	0117	Y
04	208	B	1,2,4-TRICHLOROBENZENE-D3	D	0183	Y
04	209	B	HEXACHLOROBENZENE-13C6	D	0292	Y
04	210	V	1,2-DICHLOROETHANE-D4	D	0067	Y
04	211	V	1,1,1-TRICHLOROETHANE-D3	D	0102	Y
04	212	B	HEXACHLOROETHANE-1-13C	D	0204	Y
04	213	V	1,1-DICHLOROETHANE-2,2,2-D3	D	0066	Y
04	214	V	1,1,2-TRICHLOROETHANE-13C2	D	0084	Y
04	215	V	1,1,2,2-TETRACHLOROETHANE-D2	D	0084	Y
04	216	V	CHLOROETHANE-D5	D	0071	Y
04	217	V		D		N
04	218	B	BIS(2-CHLOROETHYL)ETHER-D8	D	0101	Y
04	219	V		D	0113	N
04	220	B	2-CHLORONAPHTHALENE-D7	D	0169	Y
04	221	A	2,4,6-TRICHLOROPHENOL-3,5-D2	D	0200	Y
04	222	A	4-CHLORO-3-METHYLPHENOL-2,6-D2	D	0109	Y
04	223	V	CHLOROFORM-13C	D	0086	Y
04	224	A	2-CHLOROPHENOL-3,4,5,6-D4	D	0132	Y
04	225	B	1,2-DICHLOROBENZENE-D4	D	0152	Y
04	226	B	1,3-DICHLOROBENZENE-D4	D	0152	Y
04	227	B	1,4-DICHLOROBENZENE-D4	D	0152	Y
04	228	B	3,3'-DICHLOROBENZIDINE-D6	D	0258	Y
04	229	V	1,1-DICHLOROETHENE-D2	D	0065	Y
04	230	V	TRANS-1,2-DICHLOROETHENE-D2	D	0065	Y
04	231	A	2,4-DICHLOROPHENOL-3,5,6-D3	D	0167	Y
04	232	V	1,2-DICHLOROPROPANE-D6	D	0067	Y
04	233	V	T-1,3-DICHLOROPROPENE-1,2-D4	D	0079	Y
04	234	B	2,4-DIMETHYLPHENOL-3,5,6-D3	D	0125	Y
04	235	B	2,4-DINITROTOLUENE-3,5,6-D3	D	0168	Y
04	236	B	2,6-DINITROTOLUENE-A,A,A-D3	D	0167	Y
04	237	B	1,2-DIPHENYL-D10-HYDRAZINE	D	0082	Y
04	238	V	ETHYLBENZENE-D10	D	0116	Y
04	239	B	FLUORANTHENE-D10	D	0212	Y
04	240	B	4-CHLOROPHENYL PHENYL-D5 ETHER	D	0209	Y
04	241	B		D		N
04	242	B	BIS(2-CHLOROISOPROPYL)ETHERD12	D	0131	Y
04	243	B		D		N
04	244	V	METHYLENE CHLORIDE-D2	D	0088	Y
04	245	V	CHLOROMETHANE-D3	D	0053	Y
04	246	V	BROMOMETHANE-D3	D	0099	Y

COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	247	V	BROMOFORM-13C	D	0176	Y
04	248	V	BROMODICHLOROMETHANE-13C	D	0086	Y
04	249	V		D		N
04	250	V		D		N
04	251	V	DIBROMOCHLOROMETHANE-13C	D	0130	Y
04	252	B	HEXACHLORO-1,3-BUTADIENE-13C4	D	0231	Y
04	253	B	HEXACHLOROCYCLOPENTADIENE-13C4	D	0241	Y
04	254	B	ISOPHORONE-D8	D	0088	Y
04	255	B	NAPHTHALENE-D8	D	0136	Y
04	256	B	NITROBENZENE-D5	D	0128	Y
04	257	A	2-NITROPHENOL-3,4,5,6-D4	D	0143	Y
04	258	A	4-NITROPHENOL-2,3,5,6-D4	D	0143	Y
04	259	A	2,4-DINITROPHENOL-3,5,6-D3	D	0187	Y
04	260	A	2-METHYL-4,6-DINITROPHENOL-D2	D	0200	Y
04	261	B		D		N
04	262	B	N-NITR[illegible]ODIPHENYLAMINE-D6	D	0175	Y
04	263	B		D		N
04	264	A	PENTAC[illegible]HENOL-13C6	D	0272	Y
04	265	B	PHENOL-[illegible]5,6-D5	D	0071	Y
04	266	B	BIS(2-E[illegible]XYL)PHTHALATE-D4	D	0153	Y
04	267	B		D		N
04	268	B	DI-N-BUTYL PHTHALATE-D4	D	0153	Y
04	269	B	DI-N-OCTYL PHTHALATE-D4	D	0153	Y
04	270	B	DIETHYL PHTHALATE-3,4,5,6-D4	D	0153	Y
04	271	B	DIMETHYL PHTHALATE-3,4,5,6-D4	D	0167	Y
04	272	B	BENZO(A)ANTHRACENE-D12	D	0240	Y
04	273	B	BENZO(A)PYRENE-D12	D	0264	Y
04	274	B	BENZO(B)FLUORANTHENE-D12	D	0264	Y
04	275	B	BENZO(K)FLUORANTHENE-D12	D	0264	Y
04	276	B	CHRYSENE-D12	D	0240	Y
04	277	B	ACENAPHTHYLENE-D8	D	0160	Y
04	278	B	ANTHRACENE-D10	D	0188	Y
04	279	B	BENZO(GHI)PERYLENE-D12	D	0288	Y
04	280	B	FLUORENE-D10	D	0176	Y
04	281	B	PHENANTHRENE-D10	D	0188	Y
04	282	B		D		N
04	283	B		D		N
04	284	B	PYRENE-D10	D	0212	Y
04	285	V	TETRACHLOROETHENE-1,2-13C2	D	0172	Y
04	286	V	TOLUENE-2,3,4,5,6-D8	D	0098	Y
04	287	V	TRICHLOROETHENE-13C2	D	0136	Y
04	288	V	VINYL CHLORIDE-D3	D	0065	Y
04	289	P	ALDRIN-13C4	D		N
04	290	P	DIELDRIN-13C4	D		N
04	291	P		D		N
04	292	P	4,4'-DDT-D8	D		N
04	293	P	4,4'-DDE-D8	D		N
04	294	P	4,4'-DDD-D8	D		N
04	295	P	ENDOSULFAN-I-D4	D		N
04	296	P		D		N

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COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	297	P		D		N
04	298	P		D		N
04	299	P		D		N
04	301	B	ACENAPHTHENE	P	0154	Y
04	302	V	ACROLEIY	P	0056	Y
04	303	V	ACRYLONITRILE	P	0053	Y
04	304	V	BENZENE	P	0078	Y
04	305	B	BENZIDINE	P	0184	Y
04	306	V	CARBON TETRACHLORIDE	P	0047	Y
04	307	V	CHLOROBENZENE	P	0112	Y
04	308	B	1,2,4-TRICHLOROBENZENE	P	0180	Y
04	309	B	HEXACHLOROBENZENE	P	0284	Y
04	310	V	1,2-DICHLOROETHANE	P	0062	Y
04	311	V	1,1,1-TRICHLOROETHANE	P	0097	Y
04	312	B	HEXACHLOROETHANE	P	0201	Y
04	313	V	1,1-DICHLOROETHANE	P	0063	Y
04	314	V	1,1,2-TRICHLOROETHANE	P	0083	Y
04	315	V	1,1,2,2-TETRACHLOROETHANE	P	0083	Y
04	316	V	CHLOROETHANE	P	0064	Y
04	317	V		P		N
04	318	B	BIS(2-CHLOROETHYL)ETHER	P	0093	Y
04	319	V		P	0106	N
04	320	B	2-CHLORONAPHTHALENE	P	0162	Y
04	321	A	2,4,6-TRICHLOROPHENOL	P	0196	Y
04	322	A	4-CHLORO-3-METHYLPHENOL	P	0107	Y
04	323	V	CHLOROFORM	P	0085	Y
04	324	A	2-CHLOROPHENOL	P	0128	Y
04	325	B	1,2-DICHLOROBENZENE	P	0146	Y
04	326	B	1,3-DICHLOROBENZENE	P	0146	Y
04	327	B	1,4-DICHLOROBENZENE	P	0146	Y
04	328	B	3,3'-DICHLOROBENZIDINE	P	0252	Y
04	329	V	1,1-DICHLOROETHENE	P	0061	Y
04	330	V	TRANS-1,2-DICHLOROETHENE	P	0061	Y
04	331	A	2,4-DICHLOROPHENOL	P	0162	Y
04	332	V	1,2-DICHLOROPROPANE	P	0063	Y
04	333	V	T-1,3-DICHLOROPROPENE	P	0075	Y
04	334	B	2,4-DIMETHYLPHENOL	P	0122	Y
04	335	B	2,4-DINITROTOLUENE	P	0165	Y
04	336	B	2,6-DINITROTOLUENE	P	0165	Y
04	337	B	1,2-DIPHENYLHYDRAZINE	P	0077	Y
04	338	V	ETHYLBENZENE	P	0106	Y
04	339	B	FLUORANTHENE	P	0202	Y
04	340	B	4-CHLOROPHENYL PHENYL ETHER	P	0204	Y
04	341	B		P		N
04	342	B	BIS (2-CHLOROISOPROPYL) ETHER	P	0121	Y
04	343	B		P		N
04	344	V	METHYLENE CHLORIDE	P	0084	Y
04	345	V	CHLOROMETHANE	P	0050	Y
04	346	V	BROMOMETHANE	P	0096	Y
04	347	V	BROMOFORM	P	0173	Y

COMPOUND CODE TABLE

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TABLE     TABLE                                         CODE
NUMBER     CODE                                      DESCRIPTION

  04      348         V BROMODICHLOROMETHANE               P 0083 Y
  04      349         V                                    P      N
  04      350         V                                    P      N
  04      351         V DIBROMOCHLOROMETHANE               P 0129 Y
  04      352         B HEXACHLORO-1,3-BUTADIENE           P 0225 Y
  04      353         B HEXACHLOROCYCLOPENTADIENE          P 0237 Y
  04      354         B ISOPHORONE                         P 0082 Y
  04      355         B NAPHTHALENE                        P 0128 Y
  04      356         B NITROBENZENE                       P 0123 Y
  04      357         A 2-NITROPHENOL                      P 0139 Y
  04      358         A 4-NITROPHENOL                      P 0139 Y
  04      359         A 2,4-DINITROPHENOL                  P 0184 Y
  04      360         A 2-METHYL-4,6-DINITROPHENOL         P 0198 Y
  04      361         B                                    P      N
  04      362         B N-NITROSODIPHENYLAMINE             P 0169 Y
  04      363         B                                    P      N
  04      364         A PENTACHLOROPHENOL                  P 0266 Y
  04      365         B PHENOL                             P 0094 Y
  04      366         B BIS (2-ETHYLHEXYL) PHTHALATE       P 0149 Y
  04      367         B                                    P      N
  04      368         B DI-N-BUTYL PHTHALATE               P 0149 Y
  04      369         B DI-N-OCTYL PHTHALATE               P 0149 Y
  04      370         B DIETHYL PHTHALATE                  P 0149 Y
  04      371         B DIMETHYL PHTHALATE                 P 0163 Y
  04      372         B BENZO(A)ANTHRACENE                 P 0228 Y
  04      373         B BENZO(A)PYRENE                     P 0252 Y
  04      374         B BENZO(B)FLUORANTHENE               P 0252 Y
  04      375         B BENZO(K)FLUORANTHENE               P 0252 Y
  04      376         B CHRYSENE                           P 0228 Y
  04      377         B ACENAPHTHYLENE                     P 0152 Y
  04      378         B ANTHRACENE                         P 0178 Y
  04      379         B BENZO(GHI)PERYLENE                 P 0276 Y
  04      380         B FLUORENE                           P 0166 Y
  04      381         B PHENANTHRENE                       P 0178 Y
  04      382         B                                    P      N
  04      383         B                                    P      N
  04      384         B PYRENE                             P 0202 Y
  04      385         V TETRACHLOROETHENE                  P 0166 Y
  04      386         V TOLUENE                            P 0092 Y
  04      387         V TRICHLOROETHENE                    P 0095 Y
  04      388         V VINYL CHLORIDE                     P 0062 Y
  04      389         P                                    P      N
  04      390         P                                    P      N
  04      391         P                                    P      N
  04      392         P                                    P      N
  04      393         P                                    P      N
  04      394         P                                    P      N
  04      395         P                                    P      N
  04      396         P                                    P      N
  04      397         P                                    P      N
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COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	398	P		P		N
04	399	P		P		N
04	400	P	HEPATACHLOR-13C4	D		N
04	402	P	ALPHA-BHC-D6	D		N
04	404	P	GAMMA-BHC-D6	D		N
04	429	D	2,3,7,8-TCDD-13C12	D	0332	Y
04	500	A	BENZOIC ACID	P		Y
04	501	A	HEXANOIC ACID	P		Y
04	502	B	B-NAPHTHYLAMINE	P	0143	Y
04	503	B	ALPHA-PICOLINE	P	0093	Y
04	504	B	DIBENZOTHIOPHENE	P	0184	Y
04	505	B	DIBENZOFURAN	P	0168	Y
04	506	B	N-DODECANE (N-C12)	P	0055	Y
04	507	B	DIPHENYLAMINE	P	0169	Y
04	508	B	DIPHENYL ETHER	P	0170	Y
04	509	B	ALPHA-TERPINEOL	P	0059	Y
04	510	B	STYRENE	P	0104	Y
04	511	B	DI-N-BUTYL AMINE	P	0086	N
04	512	B	BIPHENYL	P	0154	Y
04	513	B	P-CYMEME	P	0119	Y
04	514	V	2-BUTANONE (MEK)	P	0072	Y
04	515	V	DIETHYL ETHER	P	0074	Y
04	516	V	ACETONE	P	0058	Y
04	517	B	N-DECANE (N-C10)	P	0055	Y
04	518	B	N-TETRADECANE (N-C14)	P	0055	Y
04	519	B	N-HEXADECANE (N-C16)	P	0055	Y
04	520	B	N-OCTADECANE (N-C18)	P	0055	Y
04	521	B	N-EICOSANE (N-C20)	P	0055	Y
04	522	B	N-DOCOSANE (N-C22)	P	0055	Y
04	523	B	N-TETRACOSANE (N-C24)	P	0055	Y
04	524	B	N-HEXACOSANE (N-C26)	P	0055	Y
04	525	B	N-OCTACOSANE (N-C28)	P	0055	Y
04	526	B	N-TRIACONTANE (N-C30)	P	0055	Y
04	527	V	P-DIOXANE	P	0088	Y
04	528	B	CARBAZOLE	P	0167	Y
04	529	B	1,2,3-TRICHLOROBENZENE	P	0180	Y
04	530	A	2,3,6-TRICHLOROPHENOL	P	0196	Y
04	531	A	2,4,5-TRICHLOROPHENOL	P	0196	Y
04	532	V	ALLYL ALCOHOL	P	0057	Y
04	533	V	CARBON DISULFIDE	P	0076	Y
04	534	V	2-CHLORO-1,3-BUTADIENE	P	0053	Y
04	535	V	CHLOROACETONITRILE	P	0075	Y
04	536	V	3-CHLOROPROPENE	P	0076	Y
04	537	V	CROTONALDEHYDE	P	0070	Y
04	538	V	1,2-DIBROMOETHANE (EDB)	P	0107	Y
04	539	V	DIBROMOMETHANE	P	0093	Y
04	540	V	TRANS-1,4-DICHLORO-2-BUTENE	P	0075	Y
04	541	V	1,3-DICHLOROPROPANE	P	0076	Y
04	542	V	CIS-1,3-DICHLOROPROPENE	P	0075	Y
04	543	V	ETHYL CYANIDE	P	0054	Y

COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	544	V	ETHYL METHACRYLATE	P	0069	Y
04	545	V	2-HEXANONE	P	0058	Y
04	546	V	IODOMETHANE	P	0142	Y
04	547	V	ISOBUTYL ALCOHOL	P	0074	Y
04	548	V	METHACRYLONITRILE	P	0067	Y
04	549	V	METHYL METHACRYLATE	P	0069	Y
04	550	V	4-METHLY-2-PENTANONE	P	0058	Y
04	551	V	1,1,1,2-TETRACHLOROETHANE	P	0131	Y
04	552	V	TRICHLOROFLUOROMETHANE	P	0101	Y
04	553	V	1,2,3-TRICHLOROPROPANE	P	0075	Y
04	554	V	VINYL ACETATE	P	0086	Y
04	555	B	ACETOPHENONE	P	0105	Y
04	556	B	4-AMINOBIPHENYL	P	0169	Y
04	557	B	ANILINE	P	0093	Y
04	558	B	O-ANISIDINE	P	0108	Y
04	559	B	ARAMITE	P	0185	Y
04	560	B	BENZANTHRONE	P	0230	Y
04	561	B	1,3-BENZENEDIOL (RESORCINOL)	P	0110	Y
04	562	B	BENZENETHIOL	P	0110	Y
04	563	B	2,3-BENZOFLUORENE	P	0216	Y
04	564	B	BENZYL ALCOHOL	P	0079	Y
04	565	B	2-BROMOCHLOROBENZENE	P	0111	Y
04	566	B	3-BROMOCHLOROBENZENE	P	0192	Y
04	567	B	4-CHLORO-2-NITROANILINE	P	0172	Y
04	568	B	5-CHLORO-O-TOLUIDINE	P	0106	Y
04	569	B	4-CHLOROANILINE	P	0127	Y
04	570	B	3-CHLORONITROBENZENE	P	0157	Y
04	571	B	O-CRESOL	P	0108	Y
04	572	B	CROTOXYPHOS	P	0127	Y
04	573	B	2,6-DI-TERT-BUTYL-P-BENZOQINO	P	0220	Y
04	574	B	2,4-DIAMINOTOLUENE	P	0122	Y
04	575	B	1,2-DIBROMO-3-CHLOROPROPANE	P	0157	Y
04	576	B	2,6-DICHLORO-4-NITROANILINE	P	0124	Y
04	577	B	1,3-DICHLORO-2-PROPANOL	P	0079	Y
04	578	B	2,3-DICHLOROANILINE	P	0161	Y
04	579	B	2,3-DICHLORONITROBENZENE	P	0191	Y
04	580	B	1,2:3,4-DIEPOXYBUTANE	P	0055	Y
04	581	B	3,3'-DIMETHOXYBENZIDINE	P	0224	Y
04	582	B	DIMETHYL SULFONE	P	0079	Y
04	583	B	P-DIMETHYLAMINOAZOBENZENE	P	0120	Y
04	584	B	7,12-DIMETHYLBENZ(A)ANTHRACENE	P	0256	Y
04	585	B	N,N-DIMETHYLFORMAMIDE	P	0073	Y
04	586	B	3,6-DIMETHYLPHENANTHRENE	P	0206	Y
04	587	B	1,3-DINITROBENZENE	P	0168	Y
04	588	B	DIPHENYLDISULFIDE	P	0218	Y
04	589	B	ETHYL METHANESULFONATE	P	0109	Y
04	590	B	ETHYLENETHIOUREA	P	0102	Y
04	591	B	ETHYNYLESTRADIOL 3-METHYL ETHE	P	0227	Y
04	592	B	HEXACHLOROPROPENE	P	0213	Y
04	593	B	2-ISOPROPYLNAPHTHALENE	P	0170	Y

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COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	594	B	ISOSAFROLE	P	0162	Y
04	595	B	LONGIFOLENE	P	0161	Y
04	596	B	MALACHITE GREEN	P	0330	Y
04	597	B	METHAPYRILENE	P	0097	Y
04	598	B	METHYL METHANESULFONATE	P	0080	Y
04	599	B	2-METHYLBENZOTHIOAZOLE	P	0149	Y
04	600	A	BENZOIC-D5 ACID	D		N
04	601	A	HEXANOIC ACID-D11	D		N
04	602	B	B-NAPHTHYLAMINE-D7	D	0150	Y
04	603	B	ALPHA-PICOLINE-D7	D	0100	Y
04	604	B	DIBENZOTHIOPHENE-D8	D	0192	Y
04	605	B	DIBENZOFURAN-D8	D	0176	Y
04	606	B	N-DODECANE-D26 (N-C12)	D	0066	Y
04	607	B	DIPHENYLAMINE-D10	D	0179	Y
04	608	B	DIPHENYL ETHER-D10	D	0180	Y
04	609	B	ALPHA-TERPINEOL-D3	D	0062	Y
04	610	B	STYRENE-2,3,4,5,6-D5	D	0109	Y
04	611	B	DI-N-BUTYL AMINE-D18	D	0096	N
04	612	B	BIPHENYL-D10	D	0164	Y
04	613	B	P-CYMENE-D14	D	0130	Y
04	614	V	2-BUTANONE-4,4,4-D3 (MEK)	D	0075	Y
04	615	V	DIETHYL ETHER-D10	D	0084	Y
04	616	V	ACETONE-D6	D	0064	Y
04	617	B	N-DECANE-D22 (N-C10)	D	0066	Y
04	618	B		D		N
04	619	B	N-HEXADECANE-D34 (N-C16)	D	0066	Y
04	620	B		D		N
04	621	B	N-EICOSANE-D42 (N-C20)	D	0066	Y
04	622	B		D		N
04	623	B	N-TETRACOSANE-D50 (N-C24)	D	0066	Y
04	624	B		D		N
04	625	B		D		N
04	626	B	N-TRIACONTANE-D62 (N-C30)	D	0066	Y
04	627	V	P-DIOXANE D8	D	0096	N
04	628	B	CARBAZOLE D8	D	0175	N
04	629	B	1,2,3-TRICHLOROBENZENE D3	D	0183	N
04	630	A	2,3,6-TRICHLOROPHENOL D2	D	0200	N
04	631	A	2,4,5-TRICHLOROPHENOL D2	D	0200	N
04	700	A	BENZOIC ACID	P		Y
04	701	A	HEXANOIC ACID	P		Y
04	702	B	B-NAPHTHYLAMINE	P	0143	Y
04	703	B	2-METHYLPYRIDINE	P	0093	Y
04	704	B	DIBENZOTHIOPHENE	P	0184	Y
04	705	B	DIBENZOFURAN	P	0168	Y
04	706	B	N-DODECANE (N-C12)	P	0055	Y
04	707	B	DIPHENYLAMINE	P	0169	Y
04	708	B	DIPHENYL ETHER	P	0170	Y
04	709	B	ALPHA-TERPINEOL	P	0059	Y
04	710	B	STYRENE	P	0104	Y
04	711	B	DI-N-BUTYL AMINE	P	0086	N

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COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	712	B	BIPHENYL	P	0154	Y
04	713	B	P-CYMENE	P	0119	Y
04	714	V	2-BUTANONE (MEK)	P	0072	Y
04	715	V	DIETHYL ETHER	P	0074	Y
04	716	V	ACETONE	P	0058	Y
04	717	B	N-DECANE (N-C10)	P	0055	Y
04	718	B		P		N
04	719	B	N-HEXADECANE (N-C16)	P	0055	Y
04	720	B		P		N
04	721	B	N-EICOSANE (N-C20)	P	0055	Y
04	722	B		P		N
04	723	B	N-TETRACOSANE (N-C24)	P	0055	Y
04	724	B		P		N
04	725	B		P		N
04	726	B	N-TRIACONTANE (N-C30)	P	0055	Y
04	727	V	P-DIOXANE	P	0088	N
04	728	B	CARBAZOLE	P	0167	N
04	729	B	1,2,3-TRICHLOROBENZENE	P	0180	N
04	730	A	2,3,6-TRICHLOROPHENOL	P	0196	N
04	731	A	2,4,5-TRICHLOROPHENOL	P	0196	N
04	829	D	2,3,7,8-TCDD	P	0320	Y
04	900	B	3-METHYLCHOLANTHRENE	P	0268	Y
04	901	B	4,4'-METHYLENEBIS(2-CHLOROANI)	P	0231	Y
04	902	B	4,5-METHYLENEPHENANTHRENE	P	0190	Y
04	903	B	1-METHYLFLUORENE	P	0180	Y
04	904	B	2-METHYLNAPHTHALENE	P	0142	Y
04	905	B	1-METHYLPHENANTHRENE	P	0192	Y
04	906	B	2-(METHYLTHIO)BENZOTHIAZOLE	P	0181	Y
04	907	B	1,5-NAPHTHALENEDIAMINE	P	0158	Y
04	908	B	1,4-NAPHTHOQUINONE	P	0158	Y
04	909	B	ALPHA-NAPHTHYLAMINE	P	0143	Y
04	910	B	5-NITRO-O-TOLUIDINE	P	0152	Y
04	911	B	2-NITROANILINE	P	0138	Y
04	912	B	3-NITROANILINE	P	0138	Y
04	913	B	4-NITROANILINE	P	0138	Y
04	914	B	4-NITROBIPHENYL	P	0199	Y
04	915	B	N-NITROSODI-N-BUTYLAMINE	P	0084	Y
04	916	B	N-NITROSODIETHYLAMINE	P	0102	Y
04	917	B	N-NITROSOMETHYLETHYLAMINE	P	0088	Y
04	918	B	N-NITROSOMETHYLPHENYLAMINE	P	0106	Y
04	919	B	N-NITROSOMORPHOLINE	P	0056	Y
04	920	B	N-NITROSOPIPERIDINE	P	0114	Y
04	921	B	PENTACHLOROBENZENE	P	0248	Y
04	922	B	PENTACHLOROETHANE	P	0117	Y
04	923	B	PENTAMETHYLBENZENE	P	0148	Y
04	924	B	PERYLENE	P	0252	Y
04	925	B	PHENACETIN	P	0108	Y
04	926	B	PHENOTHIAZINE	P	0199	Y
04	927	B	1-PHENYLNAPHTHALENE	P	0204	Y
04	928	B	2-PHENYLNAPHTHALENE	P	0204	Y

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               COMPOUND CODE TABLE

 TABLE      TABLE                                          CODE
 NUMBER     CODE                                        DESCRIPTION

   04     929          B PRONAMIDE                            P 0173 Y
   04     930          B PYRIDINE                             P 0079 Y
   04     931          B SAFROLE                              P 0162 Y
   04     932          B SQUALENE                             P 0069 Y
   04     933          B 1,2,4,5-TETRACHLOROBENZENE           P 0216 Y
   04     934          B THIANAPHTHENE                        P 0134 Y
   04     935          B THIOACETAMIDE                        P 0075 Y
   04     936          B THIOXANTHONE                         P 0212 Y
   04     937          B O-TOLUIDINE                          P 0106 Y
   04     938          B 1,2,3-TRIMETHOXYBENZENE              P 0168 Y
   04     939          B 2,4,5-TRIMETHYLANILINE               P 0120 Y
   04     940          B TRIPHENYLENE                         P 0228 Y
   04     941          B TRIPROPYLENEGLYCOL METHYL ETHE       P 0059 Y
   04     942          B 1,3,5-TRITHIANE                      P 0138 Y
   04     943          A BENZOIC ACID                         P 0105 Y
   04     944          A P-CRESOL                             P 0108 Y
   04     945          A 3,5-DIBROMO-4-HYDROXYBENZONITR       P 0227 Y
   04     946          A 2,6-DICHLOROPHENOL                   P 0166 Y
   04     947          A HEXANOIC ACID                        P 0060 Y
   04     948          A 2,3,4,6-TETRACHLOROPHENOL            P 0232 Y

    TABLE COUNT:      570
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ATTACHMENT 7

ANALYTICAL METHODS 1624C AND 1625C

18 December 1986

Method 1624, Revision C

Volatile Organic Compounds by Isotope Dilution GCMS

1 Scope and application

1.1 This method is designed to determine the volatile toxic organic pollutants associated with the 1976 Consent Decree; the Resource Conservation and Recovery Act; the Comprehensive Environmental Response, Compensation and Liabilities Act; and other compounds amenable to purge and trap gas chromatography-mass spectrometry (GCMS).

1.2 The chemical compounds listed in tables 1 and 2 may be determined in waters, soils, and municipal sludges by this method. The method is designed to meet the survey requirements of the Environmental Protection Agency.

1.3 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations. The levels in table 3 typify the minimum quantity that can be detected with no interferences present.

1.4 The GCMS portions of this method are for use only by analysts experienced with GCMS or under the close supervision of such qualified persons. Laboratories unfamiliar with analyses of environmental samples by GCMS should run the performance tests in reference 1 before beginning.

2 Summary of method

2.1 The percent solids content of the sample is determined. If the solids content is known or determined to be less than one percent, stable isotopically labeled analogs of the compounds of interest are added to a 5 mL sample and the sample is purged with an inert gas at 20 - 25 °C in a chamber designed for soil or water samples. If the solids content is greater than one percent, five mL of reagent water and the labeled compounds are added to a 5 gram aliquot of sample and the mixture is purged at 40 °C. Compounds that will not purge at 20 - 25 °C or at 40 °C are purged at 75 - 85 °C.

In the purging process, the volatile compounds are transferred from the aqueous phase into the gaseous phase where they are passed into a sorbent column and trapped. After purging is completed, the trap is backflushed and heated rapidly to desorb the compounds into a gas chromatograph (GC). The compounds are separated by the GC and detected by a mass spectrometer (MS) (references 2 and 3). The labeled compounds serve to correct the variability of the analytical technique.

2.2 Identification of a pollutant (qualitative analysis) is performed in one of three ways: (1) For compounds listed in table 1 and other compounds for which authentic standards are available, the GCMS system is calibrated and the mass spectrum and retention time for each standard are stored in a user created library. A compound is identified when its retention time and mass spectrum agree with the library retention time and spectrum. (2) For compounds listed in table 2 and other compounds for which standards are not available, a compound is identified when the retention time and mass spectrum agree with those specified in this method. (3) For chromatographic peaks which are not identified by (1) and (2) above, the background corrected spectrum at the peak maximum is compared with spectra in the EPA/NIH Mass Spectral File (reference 4). Tentative identification is established when the spectrum agrees.

2.3 Quantitative analysis is performed in one of four ways by GCMS using extracted ion current profile (EICP) areas: (1) For compounds listed in table 1 and other compounds for which standards and labeled analogs are available, the GCMS system is calibrated and the compound concentration is determined using an isotope dilution technique. (2) For compounds listed in table 1 and for other compounds for which authentic standards but no labeled compounds are available, the GCMS system is calibrated and the compound concentration is determined using an internal standard technique. (3) For compounds listed in table 2 and other compounds for which standards are not available, compound concentrations are determined using known response factors. (4) For compounds for which neither standards nor known response factors are available, compound concentration is determined using the sum of the EICP areas relative to the sum of the EICP areas of the nearest eluted internal standard.

2.4 Quality is assured through reproducible calibration and testing of the purge and trap and GCMS systems.

3 Contamination and interferences

3.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing upstream of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system is demonstrated to be free from interferences under conditions of the analysis by analyzing reagent water blanks initially and with each sample batch (samples analyzed on the same

8 hr shift), as described in section 8.5.

3.2 Samples can be contaminated by diffusion of volatile organic compounds (particularly methylene chloride) through the bottle seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination.

3.3 Contamination by carry-over can occur when high level and low level samples are analyzed sequentially. To reduce carry-over, the purging device (figure 1 for samples containing less than one percent solids; figure 2 for samples containing one percent solids or greater) is cleaned or replaced with a clean purging device after each sample is analyzed. When an unusually concentrated sample is encountered, it is followed by analysis of a reagent water blank to check for carry-over. Purging devices are cleaned by washing with soap solution, rinsing with tap and distilled water, and drying in an oven at 100-125 °C. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

3.4 Interferences resulting from samples will vary considerably from source to source, depending on the diversity of the site being sampled.

4 Safety

4.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in references 5 - 7.

4.2 The following compounds covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled.

5 Apparatus and materials

5.1 Sample bottles for discrete sampling

5.1.1 Bottle--25 to 40 mL with screw cap (Pierce 13075, or equivalent). Detergent wash, rinse with tap and distilled water, and dry at >105 °C for one hr minimum before use.

5.1.2 Septum--Teflon-faced silicone (Pierce 12722, or equivalent), cleaned as above and baked at 100 - 200 °C for one hour minimum.

5.2 Purge and trap device--consists of purging device, trap, and desorber.

5.2.1 Purging devices for water and soil samples

5.2.1.1 Purging device for water samples--designed to accept 5 mL samples with water column at least 3 cm deep. The volume of the gaseous head space between the water and trap shall be less than 15 mL. The purge gas shall be introduced less than 5 mm from the base of the water column and shall pass through the water as bubbles with a diameter less than 3 mm. The purging device shown in figure 1 meets these criteria.

5.2.1.2 Purging device for solid samples--designed to accept 5 grams of solids plus 5 mL of water. The volume of the gaseous head space between the water and trap shall be less than 25 mL. The purge gas shall be introduced less than 5 mm from the base of the sample and shall pass through the water as bubbles with a diameter less than 3 mm. The purging device shall be capable of operating at ambient temperature (20 - 25 °C) and of being controlled at temperatures of 40 +/- 2 °C and 80 +/- 5 °C while the sample is being purged. The purging device shown in figure 2 meets these criteria.

5.2.2 Trap--25 to 30 cm x 2.5 mm i.d. minimum, containing the following:

5.2.2.1 Methyl silicone packing--one +/- 0.2 cm, 3 percent OV-1 on 60/80 mesh Chromosorb W, or equivalent.

5.2.2.2 Porous polymer--15 +/- 1.0 cm, Tenax GC (2,6-diphenylene oxide polymer), 60/80 mesh, chromatographic grade, or equivalent.

5.2.2.3 Silica gel--8 +/- 1.0 cm, Davison Chemical, 35/60 mesh, grade 15, or equivalent. The trap shown in figure 3 meets these specifications.

5.2.4 Desorber--shall heat the trap to 175 +/- 5 °C in 45 seconds or less. The polymer section of the trap shall not exceed a temperature of 180 °C and the remaining sections shall not exceed 220 °C during desorb, and no portion of the trap shall exceed 225 °C during bakeout. The desorber shown in figure 4 meets these specifications.

5.2.5 The purge and trap device may be a separate unit or coupled to a GC as shown in figures 4 and 5.

5.3 Gas chromatograph--shall be linearly temperature programmable with initial and final holds, shall contain a glass jet separator as the MS interface, and shall produce results which meet the calibration (section 7), quality assurance (section 8), and performance tests (section 11) of this method.

5.3.1 Column--2.8 +/- 0.4 m x 2 +/- 0.5 mm i.d. glass, packed with one percent SP-1000 on Carbopak B, 60/80 mesh, or equivalent.

5.4 Mass spectrometer--70 eV electron impact ionization; shall repetitively scan from 20 to 250 amu every 2-3 seconds, and produce a unit resolution (valleys between m/z 174-176 less than 10 percent of the height of the m/z 175 peak), background corrected mass spectrum from 50 ng 4-bromofluorobenzene (BFB) injected into the GC. The BFB spectrum shall meet the mass-intensity criteria in table 4. All portions of the GC column, transfer lines, and separator which connect the GC column to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds.

5.5 Data system--shall collect and record MS data, store mass-intensity data in spectral libraries, process GCMS data and generate reports, and shall calculate and record response factors.

5.5.1 Data acquisition--mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device.

5.5.2 Mass spectral libraries--user created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GCMS runs for the compounds of interest (section 7.2).

5.5.3 Data processing--the data system shall be used to search, locate, identify, and quantify the compounds of interest in each GCMS analysis. Software routines shall be employed to compute retention times and EICP areas. Displays of spectra, mass chromatograms, and library comparisons are required to verify results.

5.5.4 Response factors and multipoint calibrations--the data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and generate multi-point calibration curves (section 7). Computations of relative standard deviation (coefficient of variation) are useful for testing calibration linearity. Statistics on initial and on-going performance shall be maintained (sections 8 and 11).

5.6 Syringes--5 mL glass hypodermic, with Luer-lok tips.

5.7 Micro syringes--10, 25, and 100 uL.

5.8 Syringe valves--2-way, with Luer ends (Teflon or Kel-F).

5.9 Syringe--5 mL, gas-tight, with shut-off valve.

5.10 Bottles--15 mL, screw-cap with Teflon liner.

5.11 Balances

5.11.1 Analytical, capable of weighing 0.1 mg.

5.11.2 Top loading, capable of weighing 10 mg.

5.12 Equipment for determining percent moisture

5.12.1 Oven, capable of being temperature controlled at 110 +/- 5 °C.

5.12.2 Dessicator

5.12.3 Beakers--50 - 100 mL

6 Reagents and standards

6.1 Reagent water--water in which the compounds of interest and interfering compounds are not detected by this method (section 11.7). It may be generated by any of the following methods:

6.1.1 Activated carbon--pass tap water through a carbon bed (Calgon Filtrasorb-300, or equivalent).

6.1.2 Water purifier--pass tap water through a purifier (Millipore Super Q, or equivalent).

6.1.3 Boil and purge--heat tap water to 90-100 °C and bubble contaminant free inert gas through it for approx one hour. While still hot, transfer the water to screw-cap bottles and seal with a

Teflon-lined cap.

6.2 Sodium thiosulfate--ACS granular.

6.3 Methanol--pesticide quality or equivalent.

6.4 Standard solutions--purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96 percent or greater, the weight may be used without correction to calculate the concentration of the standard.

6.5 Preparation of stock solutions--prepare in methanol using liquid or gaseous standards per the steps below. Observe the safety precautions given in section 4.

6.5.1 Place approx 9.8 mL of methanol in a 10 mL ground glass stoppered volumetric flask. Allow the flask to stand unstoppered for approx 10 minutes or until all methanol wetted surfaces have dried. In each case, weigh the flask, immediately add the compound, then immediately reweigh to prevent evaporation losses from affecting the measurement.

6.5.1.1 Liquids--using a 100 uL syringe, permit 2 drops of liquid to fall into the methanol without contacting the neck of the flask. Alternatively, inject a known volume of the compound into the methanol in the flask using a micro-syringe.

6.5.1.2 Gases (chloromethane, bromomethane, chloroethane, vinyl chloride)--fill a valved 5 mL gas-tight syringe with the compound. Lower the needle to approx 5 mm above the methanol meniscus. Slowly introduce the compound above the surface of the meniscus. The gas will dissolve rapidly in the methanol.

6.5.2 Fill the flask to volume, stopper, then mix by inverting several times. Calculate the concentration in mg/mL (ug/uL) from the weight gain (or density if a known volume was injected).

6.5.3 Transfer the stock solution to a Teflon sealed screw-cap bottle. Store, with minimal headspace, in the dark at -10 to -20 °C.

6.5.4 Prepare fresh standards weekly for the gases and 2-chloroethylvinyl ether. All other standards are replaced after one month, or sooner if comparison with check standards indicate a change in concentration. Quality control check standards that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

6.6 Labeled compound spiking solution--from stock standard solutions prepared as above, or from mixtures, prepare the spiking solution to contain a concentration such that a 5-10 uL spike into each 5 mL sample, blank, or aqueous standard analyzed will result in a concentration of 20 ug/L of each labeled compound. For the gases and for the water soluble compounds (acrolein, acrylonitrile, acetone, diethyl ether, and MEK), a concentration of 100 ug/L may be used. Include the internal standards (section 7.5) in this solution so that a concentration of 20 ug/L in each sample, blank, or aqueous standard will be produced.

6.7 Secondary standards--using stock solutions, prepare a secondary standard in methanol to contain each pollutant at a concentration of 500 ug/mL. For the gases and water soluble compounds (section 6.6), a concentration of 2.5 mg/mL may be used.

6.7.1 Aqueous calibration standards--using a 25 uL syringe, add 20 uL of the secondary standard (section 6.7) to 50, 100, 200, 500, and 1000 mL of reagent water to produce concentrations of 200, 100, 50, 20, and 10 ug/L, respectively. If the higher concentration standard for the gases and water soluble compounds was chosen (section 6.6), these compounds will be at concentrations of 1000, 00, 250, 100, and 50 ug/L in the aqueous calibration standards.

6.7.2 Aqueous performance standard--an aqueous standard containing all pollutants, internal standards, labeled compounds, and BFB is prepared daily, and analyzed each shift to demonstrate performance (section 11). This standard shall contain either 20 or 100 ug/L of the labeled and pollutant gases and water soluble compounds, 10 ug/L BFB, and 20 ug/L of all other pollutants, labeled compounds, and internal standards. It may be the nominal 20 ug/L aqueous calibration standard (section 6.7.1).

6.7.3 A methanolic standard containing all pollutants and internal standards is prepared to demonstrate recovery of these compounds when syringe injection and purge and trap analyses are compared. This standard shall contain either 100 ug/mL or 500 ug/mL of the gases and water soluble compounds, and 100 ug/mL of the remaining pollutants and internal standards (consistent with the amounts in the aqueous performance standard in 6.7.2).

6.7.4 Other standards which may be needed are those for test of BFB performance (section 7.1) and for collection of mass spectra for storage in spectral libraries (section 7.2).

7 Calibration

Calibration of the GCMS system is performed by purging the compounds of interest and their labeled analogs from reagent water at the temperature to be used for analysis of samples.

7.1 Assemble the gas chromatographic apparatus and establish operating conditions given in table 3. By injecting standards into the GC, demonstrate that the analytical system meets the minimum levels in table 3 for the compounds for which calibration is to be performed, and the mass-intensity criteria in table 4 for 50 ng BFB.

7.2 Mass spectral libraries--detection and identification of the compounds of interest are dependent upon the spectra stored in user created libraries.

7.2.1 For the compounds in table 1 and other compounds for which the GCMS is to be calibrated, obtain a mass spectrum of each pollutant and labeled compound and each internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components. That only a single compound is present is determined by examination of the spectrum. Fragments not attributable to the compound under study indicate the presence of an interfering compound. Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum. An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak. Software algorithms designed to "enhance" the spectrum may eliminate distortion, but may also eliminate authentic m/z's or introduce other distortion.

7.2.3 The authentic reference spectrum is obtained under BFB tuning conditions (section 7.1 and table 4) to normalize it to spectra from other instruments.

7.2.4 The spectrum is edited by saving the 5 most intense mass spectral peaks and all other mass spectral peaks greater than 10 percent of the base peak. The spectrum may be further edited to remove common interfering masses. If 5 mass spectral peaks cannot be obtained under the scan conditions given in section 5.4, the mass spectrometer may be scanned to an m/z lower than 20 to gain additional spectral information. The spectrum obtained is stored for reverse search and for compound confirmation.

7.2.5 For the compounds in table 2 and other compounds for which the mass spectra, quantitation m/z's, and retention times are known but the instrument is not to be calibrated, add the retention time and reference compound (table 3); the response factor and the quantitation m/z (table 5); and spectrum (Appendix A) to the reverse search library. Edit the spectrum per section 7.2.4, if necessary.

7.3 Assemble the purge and trap device. Pack the trap as shown in figure 3 and condition overnight at 170 - 180 °C by backflushing with an inert gas at a flow rate of 20 - 30 mL/min. Condition traps daily for a minimum of 10 minutes prior to use.

7.3.1 Analyze the aqueous performance standard (section 6.7.2) according to the purge and trap procedure in section 10. Compute the area at the primary m/z (table 5) for each compound. Compare these areas to those obtained by injecting one uL of the methanolic standard (section 6.7.3) to determine compound recovery. The recovery shall be greater than 20 percent for the water soluble compounds (section 6.6), and 60 - 110 percent for all other compounds. This recovery is demonstrated initially for each purge and trap GCMS system. The test is repeated only if the purge and trap or GCMS systems are modified in any way that might result in a change in recovery.

7.3.2 Demonstrate that 100 ng toluene (or toluene-d8) produces an area at m/z 91 (or 99) approx one-tenth that required to exceed the linear range of the system. The exact value must be determined by experience for each instrument. It is used to match the calibration range of the instrument to the analytical range and detection limits required.

7.4 Calibration by isotope dilution--the isotope dilution approach is used for the purgeable organic compounds when appropriate labeled compounds are available and when interferences do not preclude the analysis. If labeled compounds are not available, or interferences are present, the internal standard method (section 7.5) is used. A calibration curve encompassing the concentration range of interest is prepared for each compound determined. The relative response (RR) vs concentration (ug/L) is plotted or computed using a linear regression. An example of a calibration curve for toluene using toluene-d8 is given in figure 6. Also shown are the +/- 10 percent error limits (dotted lines). Relative response is determined according to the procedures described below. A minimum of five data points are required for calibration (section 7.4.4).

7.4.1 The relative response (RR) of pollutant to labeled compound is determined from isotope ratio values calculated from acquired data. Three isotope ratios are used in this process:

R_x = the isotope ratio measured in the pure pollutant (figure 7A).

R_y = the isotope ratio of pure labeled compound (figure 7B).

R_m = the isotope ratio measured in the analytical mixture of the pollutant and labeled compounds (figure 7C)

The correct way to calculate RR is:

$RR = (R_y - R_m)(R_x + 1)/(R_m - R_x)(R_y + 1)$

If R_m is not between $2R_y$ and $0.5R_x$, the method does not apply and the sample is analyzed by the internal standard method (section 7.5).

7.4.2 In most cases, the retention times of the pollutant and labeled compound are the same and isotope ratios (R's) can be calculated from the EICP areas, where:

$R = (\text{area at } m_1/z)/(\text{area at } m_2/z)$

If either of the areas is zero, it is assigned a value of one in the calculations; that is, if: area of $m_1/z = 50721$, and area of $m_2/z = 0$, then $R = 50721/1 = 50720$. The m/z's are always selected such that $R_x > R_y$.

When there is a difference in retention times (RT) between the pollutant and labeled compounds, special precautions are required to determine the isotope ratios. R_x, R_y, and R_m are defined as follows:

$R_x = [\text{area } m_1/z \text{ (at } RT_1)]/1$

$R_y = 1/[\text{area } m_2/z \text{ (at } RT_2)]$

$R_m = [\text{area } m_1/z \text{ (at } RT_1)]/[\text{area } m_2/z \text{ (at } RT_2)]$

7.4.3 An example of the above calculations can be taken from the data plotted in figure 6 for toluene and toluene-d8. For these data, $R_x = 168920/1 = 168900$, $R_y = 1/60960 = 0.00001640$, and $R_m = 96868/82508 = 1.174$. The RR for the above data is then calculated using the equation given in section 7.4.1. For the example, RR = 1.174.

Not all labeled compounds elute before their pollutant analogs.

7.4.4 To calibrate the analytical system by isotope dilution, analyze a 5 mL aliquot of each of the aqueous calibration standards (section 6.7.1) spiked with an appropriate constant amount of the labeled compound spiking solution (section 6.6), using the purge and trap procedure in section 10. Compute the RR at each concentration.

7.4.5 Linearity--if the ratio of relative response to concentration for any compound is constant (less than 20 percent coefficient of variation) over the 5 point calibration range, an averaged relative response/concentration ratio may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range.

7.5 Calibration by internal standard--used when criteria for isotope dilution (section 7.4) cannot be met. The method is applied to pollutants having no labeled analog and to the labeled compounds. The internal standards used for volatiles analyses are bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane. Concentrations of the labeled compounds and pollutants without labeled analogs are computed relative to the nearest eluted internal standard, as shown in tables 3 and 5.

7.5.1 Response factors--calibration requires the determination of response factors (RF) which are defined by the following equation:

$RF = (A_s \times C_{is})/(A_{is} \times C_s)$, where

A_s is the EICP area at the characteristic m/z for the compound in the daily standard.

A_{is} is the EICP area at the characteristic m/z for the internal standard.

C_{is} is the concentration (ug/L) of the internal standard.

C_s is the concentration of the pollutant in the daily standard.

7.5.2 The response factor is determined at 10, 20, 50, 100, and 200 ug/L for the pollutants (optionally at five times these concentrations for gases and water soluble pollutants--see section 6.7), in a way analogous to that for calibration by isotope dilution (section 7.4.4). The RF is plotted against concentration for each compound in the standard (C_s) to produce a calibration curve.

7.5.3 Linearity--if the response factor (RF) for any compound is constant (less than 35 percent coefficient of variation) over the 5 point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point range.

7.6 Combined calibration--by adding the isotopically labeled compounds and internal standards (section 6.6) to the aqueous calibration standards (section 6.7.1), a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods. These curves are verified each shift (section 11.5) by purging the aqueous performance standard (section 6.7.2). Recalibration is required only if calibration and on-going performance (section 11.5) criteria cannot be met.

7.7 Elevated purge temperature calibration--samples containing greater than one percent solids are analyzed at a temperature of 40 +/- 2 ^{o}C (section 10). For these samples, the analytical system may be calibrated using a purge temperature of 40 +/- 2 ^{o} C in order to more closely approximate the behavior of the compounds of interest in high solids samples.

8 Quality assurance/quality control

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program (reference 8). The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

8.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in section 8.2.

8.1.2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met. Each time a modification is made to the method, the analyst is required to repeat the procedure in section 8.2 to demonstrate method performance.

8.1.3 Analyses of blanks are required to demonstrate freedom from contamination and that the compounds of interest and interfering compounds have not been carried over from a previous analysis (section 3). The procedures and criteria for analysis of a blank are described in sections 8.5.

8.1.4 The laboratory shall spike all samples with labeled compounds to monitor method performance. This test is described in section 8.3. When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits (section 14.2).

8.1.5 The laboratory shall, on an on-going basis, demonstrate through the analysis of the aqueous performance standard (section 6.7.2) that the analysis system is in control. This procedure is described in sections 11.1 and 11.5.

8.1.6 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in sections 8.4 and 11.5.2.

8.2 Initial precision and accuracy--to establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations for compounds to be calibrated:

8.2.1 Analyze two sets of four 5-mL aliquots (8 aliquots total) of the aqueous performance standard (section 6.7.2) according to the method beginning in section 10.

8.2.2 Using results of the first set of four analyses in section 8.2.1, compute the average recovery (X) in ug/L and the standard deviation of the recovery (s) in ug/L for each compound, by isotope dilution for pollutants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog.

8.2.3 For each compound, compare s and X with the corresponding limits for initial precision and accuracy found in table 6. If s and X for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, system performance is unacceptable for that compound.

NOTE: The large number of compounds in table 6 present a substantial probability that one or more will fail one of the acceptance criteria when all compounds are analyzed. To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows:

8.2.4 Using the results of the second set of four analyses, compute s and X for only those compounds which failed the test of the first set of four analyses (section 8.2.3). If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin. If, however, any of the same compounds fail again, the analysis system is not performing properly for the compound (s) in question. In this event, correct the problem and repeat the entire test (section 8.2.1).

8.3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix.

8.3.1 Spike and analyze each sample according to the method beginning in section 10.

8.3.2 Compute the percent recovery (P) of the labeled compounds using the internal standard method (section 7.5).

8.3.3 Compare the percent recovery for each compound with the corresponding labeled compound recovery limit in table 6. If the recovery of any compound falls outside its warning limit, method performance is unacceptable for that compound in that sample. Therefore, the sample matrix is complex and the sample is to be diluted and reanalyzed, per section 14.2.

8.4 As part of the QA program for the laboratory, method accuracy for wastewater samples shall be assessed and records shall be maintained. After the analysis of five wastewater samples for which the labeled compounds pass the tests in section 8.3.3, compute the average percent recovery (P) and the standard deviation of the percent recovery (s_p) for the labeled compounds only. Express the accuracy assessment as a percent recovery interval from $P - 2s_p$ to $P + 2s_p$. For example, if P = 90% and s_p = 10%, the accuracy interval is expressed as 70 - 110%. Update the accuracy assessment for each compound on a regular basis (e.g. after each 5 - 10 new accuracy measurements).

8.5 Blanks--reagent water blanks are analyzed to demonstrate freedom from carry-over (section 3) and contamination.

8.5.1 The level at which the purge and trap system will carry greater than 5 ug/L of a pollutant of interest (tables 1 and 2) into a succeeding blank shall be determined by analyzing successively larger concentrations of these compounds. When a sample contains this concentration or more, a blank shall be analyzed immediately following this sample to demonstrate no carry-over at the 5 ug/L level.

8.5.2 With each sample lot (samples analyzed on the same 8 hr shift), a blank shall be analyzed immediately after analysis of the aqueous performance standard (section 11.1) to demonstrate freedom from contamination. If any of the compounds of interest (tables 1 and 2) or any potentially interfering compound is found in a blank at greater than 10 ug/L (assuming a response factor of 1 relative to the nearest eluted internal standard for compounds not listed in tables 1 and 2), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

8.6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The standards used for calibration (section 7), calibration verification (section 11.5) and for initial (section 8.2) and on-going (section 11.5) precision and accuracy should be identical, so that the most precise results will be obtained. The GCMS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analyses of volatiles by this method.

8.7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when the internal method is used.

9 Sample collection, preservation, and handling

9.1 Grab samples are collected in glass containers having a total volume greater than 20 mL. For aqueous samples which pour freely, fill sample bottles so that no air bubbles pass through the sample as the bottle is filled and seal each bottle so that no air bubbles are entrapped. Maintain the hermetic seal on the sample bottle until time of analysis.

9.2 Samples are maintained at 0 - 4 °C from the time of collection until analysis. If an aqueous sample contains residual chlorine, add sodium thiosulfate preservative (10 mg/40 mL) to the empty sample bottles just prior to shipment to the sample site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine (reference 9). If preservative has been added, shake the bottle vigorously for one minute immediately after filling.

9.3 For aqueous samples, experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions. Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. For this reason, a separate sample should be collected, acidified, and analyzed when these aromatics are to be determined. Collect about 500 mL of sample in a clean container. Adjust the pH of the sample to about 2 by adding HCl (1+1) while stirring. Check pH with narrow range (1.4 to 2.8) pH paper. Fill a sample container as described in section 9.1. If residual chlorine is present, add sodium thiosulfate to a separate sample container and fill as in section 9.1.

9.4 All samples shall be analyzed within 14 days of collection.

10 Purge, trap, and GCMS analysis--samples containing less than one percent solids are analyzed directly as aqueous samples (section 10.4). Samples containing greater one percent solids or greater are analyzed as solid samples (section 10.5).

10.1 Determination of percent solids

10.1.1 Weigh 5 - 10 g of sample into a tared beaker.

10.1.2 Dry overnight (12 hours minimum) at 110 +/- 5 ^{o}C, and cool in a dessicator.

10.1.3 Determine percent solids as follows:

$$\% \text{ solids} = \frac{\text{weight of sample dry}}{\text{weight of sample wet}} \times 100$$

10.2 Remove standards and samples from cold storage and bring to 20 - 25 °C.

10.3 Adjust the purge gas flow rate to 40 +/- 4 mL/min.

10.4 Samples containing less than one percent solids

10.4.1 Mix the sample by shaking vigorously. Remove the plunger from a 5 mL syringe and attach a closed syringe valve. Open the sample bottle and carefully pour the sample into the syringe barrel until it overflows. Replace the plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 +/- 0.1 mL. Because this process of taking an aliquot destroys the validity of the sample for future analysis, fill a second syringe at this time to protect against possible loss of data.

10.4.2 Add an appropriate amount of the labeled compound spiking solution (section 6.6) through the valve bore, then close the valve.

10.4.3 Attach the syringe valve assembly to the syringe valve on the purging device. Open both syringe valves and inject the sample into the purging chamber. Purge the sample per section 10.6.

10.5 Weighing of samples containing one percent solids or greater.

10.5.1 Mix the sample thoroughly using a clean spatula.

10.5.2 Weigh 5 +/- 1 grams of sample into a purging vessel (figure 2). Record the weight to three significant figures.

10.5.3 Add 5.0 +/- 0.1 mL of reagent water to the vessel.

10.5.4 Using a metal spatula, break up any lumps of sample to disperse the sample in the water.

10.5.5 Add an appropriate amount of the labeled compound spiking solution (section 6.6) to the sample in the purge vessel. Place a cap on the purging vessel and and shake vigorously to further disperse the sample. Attach the purge vessel to the purging device.

10.6 Purge the sample for 11.0 +/- 0.1 minutes at 20 - 25 °C for samples containing less than one percent solids. Purge samples containing one percent solids or greater at 40 +/- 2 °C. If the compounds in table 2 that do not purge at 20 - 40 °C are to be determined, a purge temperature of 80 +/- 5 °C is used.

10.7 After the 11 minute purge time, attach the trap to the chromatograph and set the purge and trap apparatus to the desorb mode (figure 5). Desorb the trapped compounds into the GC column by heating the trap to 170 -180 °C while backflushing with carrier gas at 20 - 60 mL/min for four minutes. Start MS data acquisition upon start of the desorb cycle, and start the GC column temperature program 3 minutes later. Table 3 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and minimum levels that can be achieved under these conditions. An example of the separations achieved by the column listed is shown in figure 9. Other columns may be used provided the requirements in section 8 are met. If the priority pollutant gases produce GC peaks so broad that the precision and recovery specifications (section 8.2) cannot be met, the column may be cooled to ambient or sub-ambient temperatures to sharpen these peaks.

10.8 After desorbing the sample for four minutes, recondition the trap by purging with purge gas while maintaining the trap temperature at 170 - 180 °C. After approximately seven minutes, turn off the trap heater to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

10.9 While analysis of the desorbed compounds proceeds, remove and clean the purge device. Rinse with tap water, clean with detergent and water, rinse with tap and distilled water, and dry for one hour minimum in an oven at a temperature greater than 150 °C.

11 System performance

11.1 At the beginning of each 8 hr shift during which analyses are performed, system calibration and performance shall be verified for the pollutants and labeled compounds (table 1). For these tests, analysis of the aqueous performance standard (section 6.7.2) shall be used to verify all performance criteria. Adjustment and/or recalibration (per section 7) shall be performed until all performance criteria are met. Only after all performance criteria are met may blanks and samples be analyzed.

11.2 BFB spectrum validity--the criteria in table 4 shall be met.

11.3 Retention times--the absolute retention times of the internal standards shall be as follows: bromochloromethane: 653 - 782 seconds; 2-bromo-1-chloropropane: 1270 - 1369 seconds; 1,4-dichlorobutane: 1510 - 1605 seconds. The relative retention times of all pollutants and labeled compounds shall fall within the limits given in table 3.

11.4 GC resolution--the valley height between toluene and toluene-d8 (at m/z 91 and 99 plotted on the same graph) shall be less than 10 percent of the taller of the two peaks.

11.5 Calibration verification and on-going precision and accuracy--compute the concentration of each pollutant (table 1) by isotope dilution (section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant (table 1) which has no labeled analog by the internal standard method (section 7.5). Compute the concentration of the labeled compounds by the internal standard method. These concentrations are computed based on the calibration data determined in section 7.

11.5.1 For each pollutant and labeled compound, compare the concentration with the corresponding limit for on-going accuracy in table 6. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may continue. If any individual value falls outside the range given, system performance is unacceptable for that compound.

NOTE: The large number of compounds in table 6 present a substantial probability that one or more will fail the acceptance criteria when all compounds are analyzed. To determine if the analytical system is out of control, or if the failure may be attributed to probability, proceed as follows:

11.5.1.1 Analyze a second aliquot of the aqueous performance standard (section 6.7.2).

11.5.1.2 Compute the concentration for only those compounds which failed the first test (section 11.5.1). If these compounds now pass, system performance is acceptable for all compounds and analyses of blanks and samples may proceed. If, however, any of the compounds fail again, the measurement system is not performing properly for these compounds. In this event, locate and correct the problem or recalibrate the system (section 7), and repeat the entire test (section 11.1) for all compounds.

11.5.2 Add results which pass the specification in 11.5.1.2 to initial (section 8.2) and previous on-going data. Update QC charts to form a graphic representation of laboratory performance (figure 8).

Develop a statement of accuracy for each pollutant and labeled compound by calculating the average percent recovery (R) and the standard deviation of percent recovery (s_r). Express the accuracy as a recovery interval from R - $2s_r$ to R + $2s_r$. For example, if R = 95% and s_r = 5%, the accuracy is 85 - 105 percent.

12 Qualitative determination--identification is accomplished by comparison of data from analysis of a sample or blank with data stored in the mass spectral libraries. For compounds for which the relative retention times and mass spectra are known, identification is confirmed per sections 12.1 and 12.2. For unidentified GC peaks, the spectrum is compared to spectra in the EPA/NIH mass spectral file per section 12.3.

12.1 Labeled compounds and pollutants having no labeled analog (tables 1 and 2):

12.1.1 The signals for all characteristic m/z's stored in the spectral library (section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

12.1.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two (0.5 to 2 times) for all masses stored in the library.

12.1.3 For the compounds for which the system has been calibrated (table 1), the relative retention time shall be within the windows specified in table 3.

12.1.4 For the compounds for which the system has not been calibrated but the relative retention times and mass spectra are known (table 2), the retention time relative to the internal standard specified in table 3 shall be within +/- 20 scans or +/- 60 seconds, whichever is greater, based on the nominal relative retention time specified in table 3.

12.2 Pollutants having a labeled analog (table 1):

12.2.1 The signals for all characteristic m/z's stored in the spectral library (section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

12.2.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library.

13.2.3 The relative retention time between the pollutant and its labeled analog shall be within the windows specified in table 3.

12.3 Unidentified GC peaks

12.3.1 The signals for m/z's specific to a GC peak shall all maximize within the same two consecutive scans.

12.3.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two with the masses stored in the EPA/NIH Mass Spectral File.

12.4 M/z's present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or background ions. If the experimental mass spectrum is contaminated, or if identification is ambiguous, an experienced spectrometrist (section 1.4) is to determine the presence or absence of the compound.

13 Quantitative determination

13.1 Isotope dilution--by adding a known amount of a labeled compound to every sample prior to purging, correction for recovery of the pollutant can be made because the pollutant and its labeled analog exhibit the same effects upon purging, desorption, and gas chromatography. Relative response (RR) values for sample mixtures are used in conjunction with calibration curves described in section 7.4 to determine concentrations directly, so long as labeled compound spiking levels are constant. For the toluene example given in figure 7 (section 7.4.3), RR would be equal to 1.174. For this RR value, the toluene calibration curve given in figure 6 indicates a concentration of 31.8 ug/L.

13.2 Internal standard--calculate the concentration using the response factor determined from calibration data (section 7.5) for the compounds which were calibrated (table 1), or from table 5 for compounds which were not calibrated (table 2), using the following equation:

$$\text{Concentration} = (A_s \times C_{is})/(A_{is} \times RF)$$

where the terms are as defined in section 7.5.1.

13.3 The concentration of the pollutant in the solid phase of the sample is computed using the concentration of the pollutant detected in the aqueous solution, as follows:

$$\text{Concentration in solid (ug/kg)} = \frac{0.005\ \text{L} \times \text{aqueous conc (ug/L)}}{0.01 \times \%\ \text{solids (g)}}$$

where "% solids" is from section 10.1.3.

13.4 If the EICP area at the quantitation m/z exceeds the calibration range of the system, the sample is diluted with reagent water by successive factors of 10 and the dilutions are analyzed until the area is within the calibration range.

13.5 For GC peaks which are to be identified (per section 12.3), the sample is diluted by successive factors of 10 when any peak in the uncorrected mass spectrum at the GC peak maximum is saturated.

13.6 Report results for all pollutants, labeled compounds, and tentatively identified compounds found in all standards, blanks, and samples, in ug/L for samples containing less than one percent solids and in ug/kg for samples in which the undilute sample contains one percent solids or greater, to three significant figures. Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation m/z is within the calibration range (section 13.4) or at which no m/z in the spectrum is saturated (section 13.5). For compounds having a labeled analog, results are reported at the least dilute level at which the area at the quantitation m/z is within the calibration range (section 13.4) and the labeled compound recovery is within the normal range for the method (section 14.2).

Y

14 Analysis of complex samples

14.1 Some samples may contain high levels (>1000 ug/kg) of the compounds of interest and of interfering compounds. Some samples will foam excessively when purged; others will overload the trap/or GC column.

14.2 Dilute 0.5 mL of samples containing less than one percent solids or 0.5 gram of samples containing one percent solids or greater with 4.5 mL of reagent water and analyze this diluted sample when the recovery of any labeled compound is outside the range given in table 6. If the recovery remains outside of the range for this diluted sample, the aqueous performance standard shall be analyzed (section 11) and calibration verified (section 11.5). If the recovery for the labeled compound in the aqueous performance standard is outside the range given in table 6, the analytical system is out of control. In this case, the instrument shall be repaired, the performance specifications in section 11 shall be met, and the analysis of the undiluted sample shall be repeated. If the recovery for the aqueous performance standard is within the range given in table 6, the method does not work on the sample being analyzed and the result may not be reported for regulatory compliance purposes.

14.3 Reverse search computer programs can misinterpret the spectrum of chromatographically unresolved pollutant and labeled compound pairs with overlapping spectra when a high level of the pollutant is present. Examine each chromatogram for peaks greater than the height of the internal standard peaks. These peaks can obscure the compounds of interest.

15 Method performance

15.1 The specifications for this method were taken from the interlaboratory validation of EPA Method 624 (reference 10). Method 1624 has been shown to yield slightly better performance on treated effluents than method 624. Results of initial tests of this method at a purge temperature of 80 °C can be found in reference 11 and results of initial tests of this method on municipal sludge can be found in reference 12.

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Table 1

Volatile Organic Compounds Determined by Calibrated GCMS using Isotope Dilution and Internal Standard Techniques

		Pollutant			Labeled Compound		
Compound	Storet	CAS Registry	EPA-EGD	NPDES	Analog	CAS Registry	EPA-EGD
acetone	81552	67-64-1	516 V		d6	666-52-4	616 V
acrolein	34210	107-02-8	002 V	001 V	d4	33984-05-3	202 V
acrylonitrile	34215	107-13-1	003 V	002 V	d3	53807-26-4	203 V
benzene	34030	71-43-2	004 V	003 V	d6	1076-43-3	204 V
bromodichloromethane	32101	75-27-4	048 V	012 V	13C	93952-10-4	248 V
bromoform	32104	75-25-2	047 V	005 V	13C	72802-81-4	247 V
bromomethane	34413	74-83-9	046 V	020 V	d3	1111-88-2	246 V
carbon tetrachloride	32102	56-23-5	006 V	006 V	13C	32488-50-9	206 V
chlorobenzene	34301	108-90-7	007 V	007 V	d5	3114-55-4	207 V
chloroethane	34311	75-00-3	016 V	009 V	d5	19199-91-8	216 V
2-chloroethylvinyl ether	34576	110-75-8	019 V	010 V			
chloroform	32106	67-66-3	023 V	011 V	13C	31717-44-9	223 V
chloromethane	34418	74-87-3	045 V	021 V	d3	1111-89-3	245 V
dibromochloromethane	32105	124-48-1	051 V	008 V	13C	93951-99-6	251 V
1,1-dichloroethane	34496	75-34-3	013 V	014 V	d3	56912-77-7	213 V
1,2-dichloroethane	32103	107-06-2	010 V	015 V	d4	17070-07-0	210 V
1,1-dichloroethene	34501	75-35-4	029 V	016 V	d2	22280-73-5	229 V
trans-1,2-dichlorethene	34546	156-60-5	030 V	026 V	d3	42366-47-2	230 V
1,2-dichloropropane	34541	78-87-5	032 V	017 V	d6	93952-08-0	232 V
trans-1,3-dichloropropene	34699	10061-02-6	033 V		d4	93951-86-1	233 V
diethyl ether	81576	60-29-7	515 V		d10	2679-89-2	615 V
p-dioxane	81582	123-91-1	527 V		d8	17647-74-4	627 V
ethylbenzene	34371	100-41-4	038 V	019 V	d10	25837-05-2	238 V
methylene chloride	34423	75-09-2	044 V	022 V	d2	1665-00-5	244 V
methyl ethyl ketone	81595	78-93-3	514 V		d3	53389-26-7	614 V
1,1,2,2-tetrachloroethane	34516	79-34-5	015 V	023 V	d2	33685-54-0	215 V
tetrachlorethene	34475	127-18-4	085 V	024 V	13C2	32488-49-6	285 V
toluene	34010	108-88-3	086 V	025 V	d8	2037-26-5	286 V
1,1,1-trichloroethane	34506	71-55-6	011 V	027 V	d3	2747-58-2	211 V
1,1,2-trichloroethane	34511	79-00-5	014 V	028 V	13C2	93952-09-1	214 V
trichloroethene	39180	79-01-6	087 V	029 V	13C2	93952-00-2	287 V
vinyl chloride	39175	75-01-4	088 V	031 V	d3	6745-35-3	288 V

Table 2

Volatile Organic Compounds to be Determined by Reverse Search and Quantitation using Known Retention Times, Response Factors, Reference Compounds, and Mass Spectra

EGD No.	Compound	CAS Registry
532	allyl alcohol*	107-18-6
533	carbon disulfide	75-15-0
534	2-chloro-1,3-butadiene (chloroprene)	126-99-8
535	chloroacetonitrile*	107-14-2
536	3-chloropropene	107-05-1
537	crotonaldehyde*	123-73-9
538	1,2-dibromoethane (EDB)	106-93-4
539	dibromomethane	74-95-3
540	trans-1,4-dichloro-2-butene	110-47-6
541	1,3-dichloropropane	142-28-9
542	cis-1,3-dichloropropene	10061-01-5
543	ethyl cyanide*	107-12-0
544	ethyl methacrylate	97-63-2
545	2-hexanone	591-78-6
546	iodomethane	74-88-4
547	isobutyl alcohol*	78-83-1
548	methacrylonitrile	126-98-7
549	methyl methacrylate	78-83-1
550	4-methyl-2-pentanone	108-10-1
551	1,1,1,2-tetrachloroethane	630-20-6
552	trichlorofluoromethane	75-69-4
553	1,2,3-trichloropropane	96-18-4
554	vinyl acetate	108-05-4

*determined at a purge temperature of 75 - 85 °C

Table 3

Gas Chromatography of Purgeable Organic Compounds

EGD No. (1)	Compound	Retention time Mean (sec)	EGD Ref	Relative (2)	Minimum level (3) (ug/L)	Method Detection Limit (4) Low solids (ug/kg)	High solids (ug/kg)
245	chloromethane-d3	147	181	0.141 - 0.270	50		
345	chloromethane	148	245	0.922 - 1.210	50	207*	13
246	bromomethane-d3	243	181	0.233 - 0.423	50		
346	bromomethane	246	246	0.898 - 1.195	50	148*	11
288	vinyl chloride-d3	301	181	0.286 - 0.501	50		
388	vinyl chloride	304	288	0.946 - 1.023	10	190*	11
216	chloroethane-d5	378	181	0.373 - 0.620	50		
316	chloroethane	386	216	0.999 - 1.060	50	789*	24
244	methylene chloride-d2	512	181	0.582 - 0.813	10		
344	methylene chloride	517	244	0.999 - 1.017	10	566*	280*
546	iodomethane	498	181	0.68			
616	acetone-d6	554	181	0.628 - 0.889	50		
716	acetone	565	616	0.984 - 1.019	50	3561*	322*
202	acrolein-d4	564	181	0.641 - 0.903(5)	50		
302	acrolein	566	202	0.984 - 1.018(5)	50	377*	18
203	acrylonitrile-d3	606	181	0.735 - 0.926	50		
303	acrylonitrile	612	203	0.985 - 1.030	50	360*	9
533	carbon disulfide	631	181	0.86			
552	trichlorofluoromethane	663	181	0.91			
543	ethyl cyanide	672	181	0.92			
229	1,1-dichloroethene-d2	696	181	0.903 - 0.976	10		
329	1,1-dichloroethene	696	229	0.999 - 1.011	10	31	5
536	3-chloropropene	696	181	0.95			
532	allyl alcohol	703	181	0.96			
181	bromochloromethane (I.S.)	730	181	1.000 - 1.000	10		
213	1,1-dichloroethane-d3	778	181	1.031 - 1.119	10		
313	1,1-dichloroethane	786	213	0.999 - 1.014	10	16	1
615	diethyl ether-d10	804	181	1.067 - 1.254	50		
715	diethyl ether	820	615	1.010 - 1.048	50	63	12
230	trans-1,2-dichloroethene-d2	821	181	1.056 - 1.228	10		
330	trans-1,2-dichloroethene	821	230	0.996 - 1.011	10	41	3
614	methyl ethyl ketone-d3	840	181	0.646 - 1.202	50		
714	methyl ethyl ketone	848	614	0.992 - 1.055	50	241*	80*
223	chloroform-13C1	861	181	1.092 - 1.322	10		
323	chloroform	861	223	0.961 - 1.009	10	21	2
535	chloroacetonitrile	884	181	1.21			
210	1,2-dichloroethane-d4	901	181	1.187 - 1.416	10		
310	1,2-dichloroethane	910	210	0.973 - 1.032	10	23	3
539	dibromomethane	910	181	1.25			
548	methacrylonitrile	921	181	1.26			
547	isobutyl alcohol	962	181	1.32			
211	1,1,1-trichloroethane-13C2	989	181	1.293 - 1.598	10		
311	1,1,1-trichloroethane	999	211	0.989 - 1.044	10	16	4
627	p-dioxane-d8	982	181	1.262 - 1.448(5)	10		
727	p-dioxane	1001	627	1.008 - 1.040(5)	10	--	140*
206	carbon tetrachloride-13C1	1018	182	0.754 - 0.805	10		
306	carbon tetrachloride	1018	206	0.938 - 1.005	10	87	9
554	vinyl acetate	1031	182	0.79			
248	bromodichloromethane-13C1	1045	182	0.766 - 0.825	10		

348	bromodichloromethane	1045	248	0.978 - 1.013	10	28	3
534	2-chloro-1,3-butadiene	1084	182	0.83			
537	crotonaldehyde	1098	182	0.84			
232	1,2-dichloropropane-d6	1123	182	0.830 - 0.880	10		
332	1,2-dichloropropane	1134	232	0.984 - 1.018	10	29	5
542	cis-1,3-dichloropropen	1138	182	0.87			
287	trichloroethene-13C2	1172	182	0.897 - 0.917	10		
387	trichloroethene	1187	287	0.991 - 1.037	10	41	2
541	1,3-dichloropropane	1196	182	0.92			
204	benzene-d6	1200	182	0.888 - 0.952	10		
304	benzene	1212	204	1.002 - 1.026	10	23	8
251	chlorodibromomethane-13C1	1222	182	0.915 - 0.949	10		
351	chlorodibromomethane	1222	251	0.989 - 1.030	10	15	2
214	1,1,2-trichloroethane-13C2	1224	182	0.922 - 0.953	10		
314	1,1,2-trichloroethane	1224	214	0.975 - 1.027	10	26	1
233	trans-1,3-dichloropropene-d4	1226	182	0.922 - 0.959	10		
333	trans-1,3-dichloropropene	1226	233	0.993 - 1.016	10	(6)*	(6)*
019	2-chloroethylvinyl ether	1278	182	0.983 - 1.026	10	122	21
538	1,2-dibromoethane	1279	182	0.98			
182	2-bromo-1-chloropropane (I.S.)	1306	182	1.000 - 1.000	10		
549	methyl methacrylate	1379	182	1.06			
247	bromoform-13C1	1386	182	1.048 - 1.087	10		
347	bromoform	1386	247	0.992 - 1.003	10	91	7
551	1,1,1,2-tetrachloroethane	1408	182	1.08			
550	4-methyl-2-pentanone	1435	183	0.92			
553	1,2,3-trichloropropane	1520	183	0.98			
215	1,1,2,2-tetrachloroethane-d2	1525	183	0.969 - 0.996	10		
315	1,1,2,2-tetrachloroethane	1525	215	0.890 - 1.016	10	20	6
545	2-hexanone	1525	183	0.98			
285	tetrachloroethene-13C2	1528	183	0.966 - 0.996	10		
385	tetrachloroethene	1528	285	0.997 - 1.003	10	106	10
540	trans-1,4-dichloro-2-butene	1551	183	1.00			
183	1,4-dichlorobutane (int std)	1555	183	1.000 - 1.000	10		
544	ethyl methacrylate	1594	183	1.03			
286	toluene-d8	1603	183	1.016 - 1.054	10		
386	toluene	1619	286	1.001 - 1.019	10	27	4
207	chlorobenzene-d5	1679	183	1.066 - 1.135	10		
307	chlorobenzene	1679	207	0.914 - 1.019	10	21	58*
238	ethylbenzene-d10	1802	183	1.144 - 1.293	10		
338	ethylbenzene	1820	238	0.981 - 1.018	10	28	4
185	bromofluorobenzene	1985	183	1.255 - 1.290	10		

(1) Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.
(2) Based on data from a single wastewater laboratory.
(3) This is a minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points when calibrated using reagent water. The concentration in the aqueous or solid phase is determined using the equations in section 13.
(4) Method detection limits determined in digested sludge (low solids) and in filter cake or compost (high solids).
(5) Specification derived from related compound.
(6) An unknown interference in the particular sludge studied precluded measurement of the Method Detection Limit (MDL) for this compound.
*Background levels of these compounds were present in the sludge resulting

in higher than expected MDL's. The MDL for these compounds is expected to be approximately 20 ug/kg (100 - 200 for the gases and water soluble compounds) for the low solids method and 5 - 10 ug/kg (25 - 50 for the gases and water soluble compounds) for the high solids method, with no interferences present.

Column: 2.4 m (8 ft) x 2 mm i.d. glass, packed with one percent SP-1000 coated on 60/80 Carbopak B.
Carrier gas: helium at 40 mL/min.
Temperature program: 3 min at 45 °C, 8 °C per min to 240 °C, hold at 240 °C for 15 minutes.
Note: The retention time specifications in this table were developed from data collected from four wastewater laboratories.

Table 4

BFB Mass-intensity Specifications

M/z	Intensity required
50	15 to 40 percent of m/z 95
75	30 to 60 percent of m/z 95
95	base peak, 100 percent
96	5 to 9 percent of m/z 95
173	less than 2 percent of m/z 174
174	greater than 50 percent of m/z 95
175	5 to 9 percent of m/z
176	95 to 100 percent of m/z 174
177	5 to 9 percent of m/z 176

Table 5

Volatile Organic Compound Characteristic M/z's

Compound	Labeled analog	Primary m/z's	Reference compound (1)	Response factor at purge temp. of 20 °C	80 °C
acetone	d6	58/64			
acrolein	d4	56/60			
acrylonitrile	d3	53/56			
allyl alcohol		57	181	(2)	0.20
benzene	d6	78/84			
2-bromo-1-chloropropane (3)		77			
bromochloromethane (3)		128			
bromodichloromethane	^{13}C	83/86			
bromoform	^{13}C	173/176			
bromomethane	d3	96/99			
carbon disulfide		76	181	1.93	2.02
carbon tetrachloride	^{13}C	47/48			
2-chloro-1,3-butadiene		53	182	0.29	0.50
chloroacetonitrile		75	181	(2)	1.12
chlorobenzene	d5	112/117			
chloroethane	d5	64/71			
2-chloroethylvinyl ether	d7	106/113			
chloroform	^{13}C	85/86			
chloromethane	d3	50/52			
3-chloropropene		76	181	0.43	0.63
crotonaldehyde		70	182	(2)	0.090
dibromochloromethane	^{13}C	129/130			
1,2-dibromoethane		107	182	0.86	0.68
dibromomethane		93	181	1.35	1.91
1,4-dichlorobutane (3)		55			
trans-1,4-dichloro-2-butene		75	183	0.093	0.14
1,1-dichloroethane	d3	63/66			
1,2-dichloroethane	d4	62/67			
1,1-dichloroethene	d2	61/65			
trans-1,2-dichlorethene	d2	61/65			
1,2-dichloropropane	d6	63/67			
1,3-dichloropropane		76	182	0.89	0.88
cis-1,3-dichloropropene		75	182	0.29	0.41
trans-1,3-dichloropropene	d4	75/79			
diethyl ether	d10	74/84			
p-dioxane	d8	88/96			
ethyl cyanide		54	181	(2)	1.26
ethyl methacrylate		69	183	0.69	0.52
ethylbenzene	d10	106/116			
2-hexanone		58	183	0.076	0.33
iodomethane		142	181	4.55	2.55
isobutyl alcohol		74	181	(2)	0.22
methylene chloride	d2	84/88			
methyl ethyl ketone	d3	72/75			
methyl methacrylate		69	182	0.23	0.79
4-methyl-2-pentanone		58	183	0.15	0.29
methacrylonitrile		67	181	0.25	0.79
1,1,1,2-tetrachloroethane		131	182	0.20	0.25
1,1,2,2-tetrachloroethane	d2	83/84			
tetrachlorethene	$^{13}C_2$	166/172			

toluene	d8	92/99			
1,1,1-trichloroethane	d3	97/102			
1,1,2-trichloroethane	^{13}C2	83/84			
trichloroethene	^{13}C2	95/136			
trichlorofluoromethane		101	181	2.31	2.19
1,2,3-trichloropropane		75	183	0.89	0.72
vinyl acetate		86	182	0.054	0.19
vinyl chloride	d3	62/65			

(1) 181 = bromochloromethane
182 = 2-bromo-1-chloropropane
183 = 1,4-dichlorobutane
(2) not detected at a purge temperature of 25 °C
(3) internal standard

Table 6

Acceptance Criteria for Performance Tests

	Acceptance criteria at 20 ug/L or as noted			
	Initial precision and accuracy Section 8.2.3		labeled compound recovery Sec 8.3 and 14.2	On-going accuracy Sec 11.5
Compound	s (ug/L)	X (ug/L)	P (%)	R (ug/L)
acetone*	51.	77 - 153	35 - 165	55 - 145
acrolein*	72.	32 - 168	37 - 163	7 - 190
acrylonitrile*	16.	70 - 132	ns - 204	58 - 144
benzene	9.0	13 - 28	ns - 196	4 - 33
bromodichloromethane	8.2	7 - 32	ns - 99	4 - 34
bromoform	7.0	7 - 35	ns - 214	6 - 36
bromomethane	25.	d - 54	ns - 414	d - 61
carbon tetrachloride	6.9	16 - 25	42 - 165	12 - 30
chlorobenzene	8.2	14 - 30	ns - 205	4 - 35
chloroethane	15.	d - 47	ns - 308	d - 51
2-chloroethylvinyl ether	36.	d - 70	ns - 554	d - 79
chloroform	7.9	12 - 26	18 - 172	8 - 30
chloromethane	26.	d - 56	ns - 410	d - 64
dibromochloromethane	7.9	11 - 29	16 - 185	8 - 32
1,1-dichloroethane	6.7	11 - 31	23 - 191	9 - 33
1,2-dichloroethane	7.7	12 - 30	12 - 192	8 - 33
1,1-dichloroethene	12.	d - 50	ns - 315	d - 52
trans-1,2-dichlorethene	7.4	11 - 32	15 - 195	8 - 34
1,2-dichloropropane	19.	d - 47	ns - 343	d - 51
cis-1,3-dichloropropene	22.	d - 51	ns - 381	d - 56
trans-1,3-dichloropropene	15.	d - 40	ns - 284	d - 44
diethyl ether*	44.	75 - 146	44 - 156	55 - 145
p-dioxane	7.2	13 - 27	ns - 239	11 - 29
ethylbenzene	9.6	16 - 29	ns - 203	5 - 35
methylene chloride	9.7	d - 50	ns - 316	d - 50
methyl ethyl ketone*	57.	66 - 159	36 - 164	42 - 158
1,1,2,2-tetrachloroethane	9.6	11 - 30	5 - 199	7 - 34
tetrachlorethene	6.6	15 - 29	31 - 181	11 - 32
toluene	6.3	15 - 29	4 - 193	6 - 33
1,1,1-trichloroethane	5.9	11 - 33	12 - 200	8 - 35
1,1,2-trichloroethane	7.1	12 - 30	21 - 184	9 - 32
trichloroethene	8.9	17 - 30	35 - 196	12 - 34
vinyl chloride	228	d - 59	ns - 452	d - 65

* Acceptance criteria at 100 ug/L
d = detected; result must be greater than zero.
ns = no specification; limit would be below detection limit.

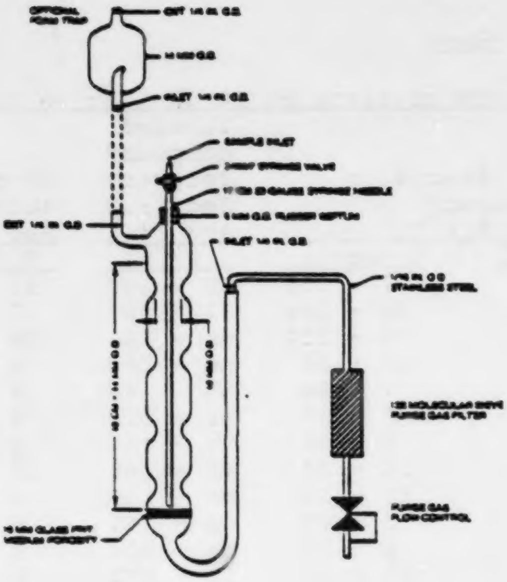


Figure 1 Purging Device for Waters

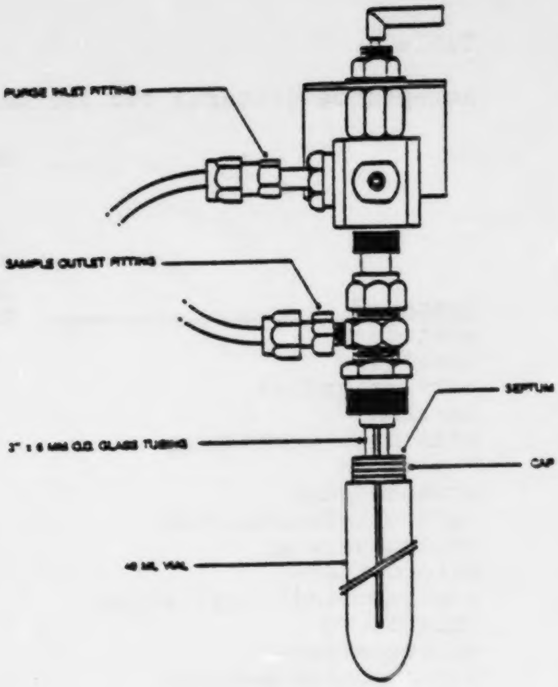


Figure 2 Purging Device for Soils or Waters

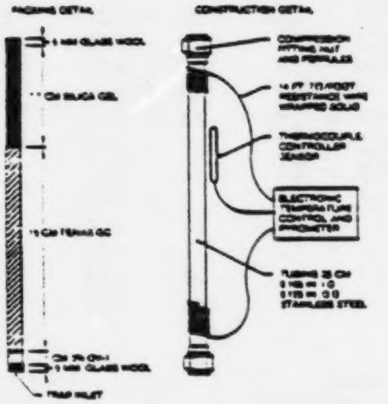

PACKING DETAIL
CONSTRUCTION DETAIL
5 MM GLASS WOOL
CM SILICA GEL
15 CM TENAX GC
1 CM 3% OV-1
5 MM GLASS WOOL
TRAP INLET
COMPRESSION FITTING NUT AND FERRULES
14 FT. 7Ω/FOOT RESISTANCE WIRE WRAPPED SOLID
THERMOCOUPLE/CONTROLLER SENSOR
ELECTRONIC TEMPERATURE CONTROL AND PYROMETER
TUBING 25 CM 0.105 IN. I.D. 0.125 IN. O.D. STAINLESS STEEL

Figure 3 Trap Construction and Packings

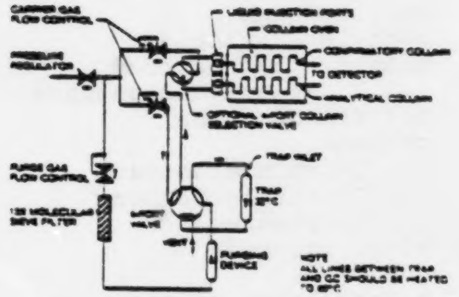


Figure 4 Schematic of Purge and Trap Device--Purge Mode

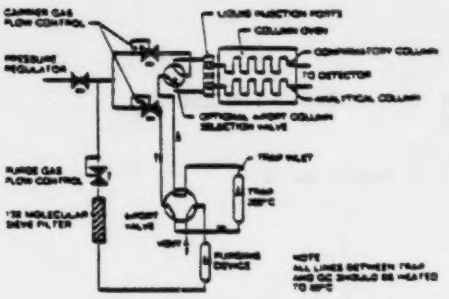


Figure 5 Schematic of Purge and Trap Device--Desorb Mode

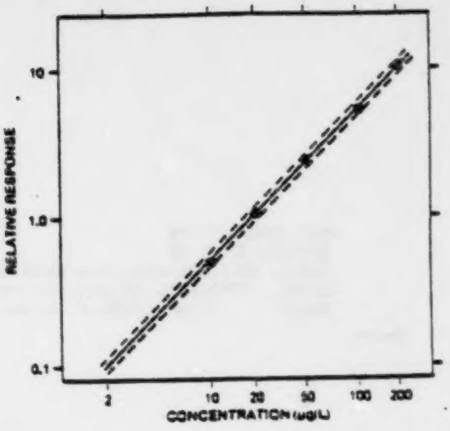


Figure 6 Relative Response Calibration Curve for Toluene. The Dotted Lines Enclose a +/- 10 Percent Error Window.

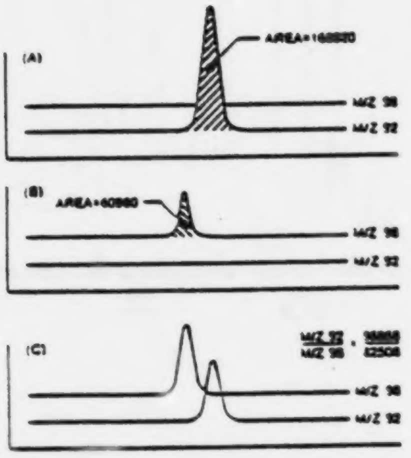


Figure 7 Extracted Ion Current Profiles for (A) Toluene, (B) Toluene-d8, and (C) a Mixture of Toluene and Toluene-d8.

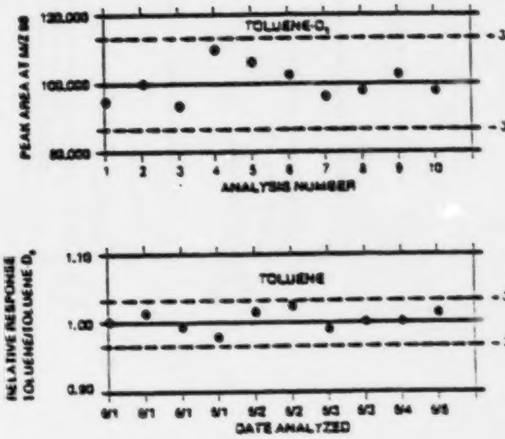
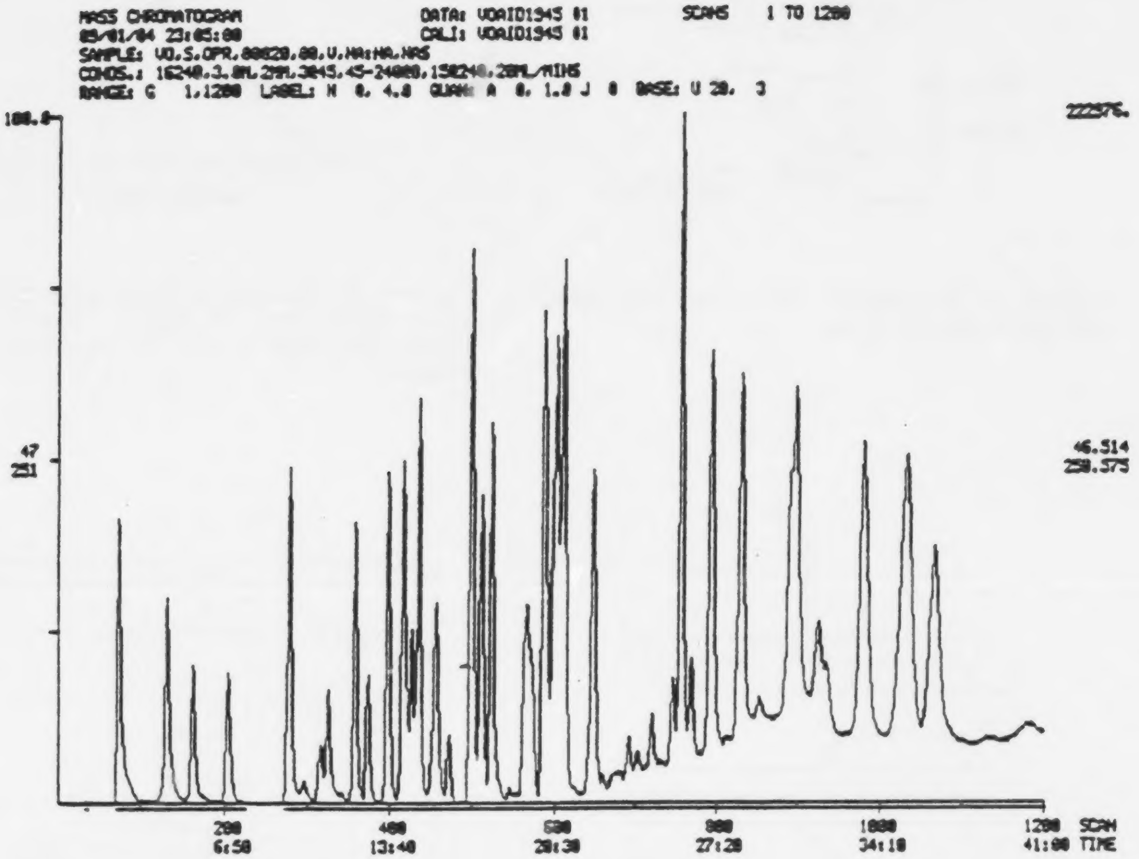


Figure 8 Quality Control Charts Showing Area (top graph) and Relative Response of Toluene to Toluene-d8 (lower graph) Plotted as a Function of Time or Analysis Number

Figure 9 Chromatogram of Aqueous Performance Standard



Appendix A: Mass Spectra in the Form of Mass/intensity Lists

532 allyl alcohol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	30	43	39	44	232	45	12	53	13	55	59
56	58	57	1000	58	300	61	15				

533 carbon disulfide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
44	282	46	10	64	14	76	1000	77	27	78	82

534 2-chloro-1,3-butadiene (chloroprene)

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
48	21	49	91	50	223	51	246	52	241	53	1000
54	41	61	30	62	54	63	11	64	16	73	21
87	12	88	452	89	22	90	137				

535 chloroacetonitrile

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
47	135	48	1000	49	88	50	294	51	12	73	22
74	43	75	884	76	39	77	278				

536 3-chloropropene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
35	39	36	40	40	44	42	206	47	40	58	35
49	176	51	64	52	31	61	29	73	22	75	138
76	1000	77	74	78	324						

537 crotonaldehyde

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
35	26	40	28	42	339	43	48	44	335	49	27
50	40	51	20	52	21	53	31	55	55	68	24
69	511	70	1000	71	43						

538 1,2-dibromoethane (EDB)

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
79	50	80	13	31	51	82	15	93	54	95	42
105	32	106	29	107	1000	108	38	109	922	110	19
186	13	188	27	190	13						

539 dibromomethane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
43	99	44	101	45	30	79	184	80	35	81	175
91	142	92	61	93	1000	94	64	95	875	160	18
172	375	173	14	174	719	175	12	176	342		

540 trans-1,4-dichloro-2-butane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	166	50	171	51	289	52	85	53	878	54	273
62	286	64	91	75	1000	77	323	88	246	89	415
90	93	91	129	124	138	126	86	128	12		

541 1,3-dichloropropane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	15	42	44	47	19	48	20	49	193	51	55
61	18	62	22	63	131	65	38	75	47	76	1000
77	46	78	310	79	12						

542 cis-1,3-dichloropropene

37	262	38	269	39	998	49	596	51	189	75	1000
77	328	110	254	112	161						

543 ethyl cyanide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
44	115	50	34	51	166	52	190	53	127	54	1000
55	193										

544 ethyl methacrylate

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	127	43	48	45	155	55	32	58	39	68	60
69	1000	70	83	71	25	85	14	86	169	87	21
96	17	99	93	113	11	114	119				

545 2-hexanone (methyl butyl ketone)

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	61	43	1000	44	24	55	12	57	130	58	382
59	21	71	36	85	37	100	56				

546 iodomethane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
44	57	127	328	128	17	139	39	140	34	141	120
142	1000	143	12								

547 isobutyl alcohol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
34	21	35	13	36	13	37	11	39	10	42	575
43	1000	44	42	45	21	55	40	56	37	57	21
59	25	73	12	74	63						

548 methacrylonitrile

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
38	24	39	21	41	26	42	100	49	19	50	60
51	214	52	446	53	19	62	24	63	59	64	136
65	55	66	400	67	1000	68	51				

549 methyl methacrylate

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	127	43	52	45	48	53	30	55	100	56	49
59	124	68	28	69	1000	70	51	82	26	85	45
98	20	99	89	100	442	101	22				

550 4-methyl-2-pentanone (methyl isobutyl ketone; MIBK)

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	69	43	1000	44	54	53	11	55	15	56	13
57	205	58	346	59	20	67	12	69	10	85	96
100	94										

551 1,1,1,2-tetrachloroethane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
47	144	49	163	60	303	61	330	62	98	82	45
84	31	95	416	96	152	97	270	98	84	117	804
121	236	131	1000	133	955	135	301				

552 trichlorofluoromethane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
44	95	47	153	49	43	51	21	52	14	66	162
68	53	82	40	84	28	101	1000	102	10	103	671
105	102	117	16	119	14						

553 1,2,3-trichloropropane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	285	51	87	61	300	62	107	63	98	75	1000
76	38	77	302	83	23	96	29	97	166	98	20
99	103	110	265	111	28	112	164	114	25		

554 vinyl acetate

36	5	42	103	43	1000	44	70	45	8	86	57

31 October 1986   Draft

Method 1625, Revision C

Semivolatile Organic Compounds by Isotope Dilution GCMS

1 Scope and application

1.1 This method is designed to determine the semivolatile toxic organic pollutants associated with the 1976 Consent Decree; the Resource Conservation and Recovery Act; the Comprehensive Environmental Response, Compensation and Liability Act; and other compounds amenable to extraction and analysis by capillary column gas chromatography-mass spectrometry (GCMS).

1.2 The chemical compounds listed in tables 1 through 4 may be determined in waters, soils, and municipal sludges by this method. The method is designed to meet the survey requirements of the Environmental Protection Agency (EPA).

1.3 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations. The limits in tables 5 and 6 typify the minimum quantity that can be detected with no interferences present.

1.4 The GCMS portions of this method are for use only by analysts experienced with GCMS or under the close supervision of such qualified persons. Laboratories unfamiliar with analyses of environmental samples by GCMS should run the performance tests in reference 1 before beginning.

2 Summary of method

2.1 The percent solids content of a sample is determined.

Stable isotopically labeled analogs of the compounds of interest are added to the sample.

If the solids content is less than one percent, a one liter sample is extracted at pH 12 - 13, then at pH <2 with methylene chloride using continuous extraction techniques. If the solids content is 30 percent percent or less, the sample is diluted to one percent solids with reagent water, homogenized ultrasonically, and extracted at pH 12-13, then at pH <2 with methylene chloride using continuous extraction techniques. If the solids content is greater than 30 percent, the sample is extracted using ultrasonic techniques.

Each extract is dried over sodium sulfate, concentrated to a volume of five mL, cleaned up using gel permeation chromatography (GPC), if necessary, and concentrated to one mL. An internal standard is added to the extract, and a one uL aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated by GC and detected by a mass spectrometer (MS). The labeled compounds serve to correct the variability of the analytical technique.

2.2 Identification of a pollutant (qualitative analysis) is performed in one of three ways: (1) For compounds listed in tables 1 and 2, and for other compounds for which authentic standards are available, the GCMS system is calibrated and the mass spectrum and retention time for each standard are stored in a user created library. A compound is identified when its retention time and mass spectrum agree with the library retention time and spectrum. (2) For compounds listed in tables 3 and 4, and for other compounds for which standards are not available, a compound is identified when the retention time and mass spectrum spectrum agree with those specified in this method. (3) For chromatographic peaks which are not identified by (1) and (2) above, the background corrected spectrum at the peak maximum is compared with spectra in the EPA/NIH Mass Spectral File (reference 2). Tentative identification is established when the spectrum agrees.

2.3 Quantitative analysis is performed in one of four ways by GCMS using extracted ion current profile (EICP) areas: (1) For compounds listed in tables 1 and 2, and for other compounds for which standards and labeled analogs are available, the GCMS system is calibrated and the compound concentration is determined using an isotope dilution technique. (2) For compounds listed in tables 1 and 2, and for other compounds for which authentic standards but no labeled compounds are available, the GCMS system is calibrated and the compound concentration is determined using an internal standard technique. (3) For compounds listed in tables 3 and 4, and for other compounds for which standards are not available, compound concentrations are determined using known response factors. (4) For compounds for which neither standards nor known response factors are available, compound concentration is determined using the sum of the EICP areas relative to the sum of the EICP areas of the internal standard.

2.4 Quality is assured through reproducible calibration and testing of the extraction and GCMS systems.

3 Contamination and interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms and spectra. All materials used in the analysis shall be demonstrated to be free from interferences under the conditions of analysis by running method blanks initially and with each sample lot (samples started through the extraction process on a given 8 hr shift, to a maximum of 20). Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Glassware and, where possible, reagents are cleaned by solvent rinse and baking at 450 °C for one hour minimum.

3.2 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled.

4 Safety

4.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in references 3 - 5.

4.2 The following compounds covered by this method have been tentatively classified as know or suspected human or mammalian carcinogens: benzo(a)anthracene, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, N-nitrosodimethylamine, and beta-naphthylamine. Primary standards of these compounds shall be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled.

5 Apparatus and materials

5.1 Sampling equipment for discrete or composite sampling.

5.1.1 Sample Bottles and Caps

5.1.1.1 Liquid Samples (waters, sludges and similar materials that contain less than five percent solids)--Sample bottle, amber glass, 1.1 liters minimum, with screw cap.

5.1.1.2 Solid samples (soils, sediments, sludges, filter cake, compost, and similar materials that contain more than five percent solids)--Sample bottle, wide mouth, amber glass, 500 mL minimum.

5.1.1.3 If amber bottles are not available, samples shall be protected from light.

5.1.1.4 Bottle caps--threaded to fit sample bottles. Caps shall be lined with Teflon.

5.1.1.5 Cleaning

5.1.1.5.1 Bottles are detergent water washed, then solvent rinsed or baked at 450 °C for one hour minimum before use.

5.1.1.5.2 Liners are detergent water washed, then reagent water (section 6.5.1) and solvent rinsed, and baked at approx 200 °C for one hour minimum prior to use.

5.1.2 Compositing equipment--automatic or manual compositing system incorporating glass containers cleaned per bottle cleaning procedure above. Sample containers are kept at 0 - 4 °C during sampling. Glass or Teflon tubing only shall be used. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used in the pump only. Before use, the tubing shall be thoroughly rinsed with methanol, followed by repeated rinsings with reagent water (section 6.5.1) to minimize sample contamination. An integrating flow meter is used to collect proportional composite samples.

5.2 Equipment for determining percent moisture

5.2.1 Oven, capable of being temperature controlled at 110 +/- 5 °C.

5.2.2 Dessicator

5.3 Sonic disruptor--375 watt with pulsing capability and 3/4 in. disruptor horn (Ultrasonics, Inc, Model 375C, or equivalent).

5.4 Extraction apparatus

5.4.1 Continuous liquid-liquid extractor--Teflon or glass connecting joints and stopcocks without lubrication, 1.5 - 2 liter capacity (Hershberg-Wolf Extractor, Ace Glass 6841-10, or equivalent).

5.4.2 Beakers

5.4.2.1 1.5 - 2 liter, calibrated to one liter

5.4.2.2 400 - 500 mL

5.4.2.3 Spatulas--stainless steel

5.4.3 Filtration apparatus

5.4.3.1 Glass funnel--125 - 250 mL

5.4.3.2 Filter paper for above (Whatman 41, or equivalent)

5.5 Drying column--15 to 20 mm i.d. Pyrex chromatographic column equipped with coarse glass frit or glass wool plug.

5.6 Kuderna-Danish (K-D) apparatus

5.6.1 Concentrator tube--10mL, graduated (Kontes K-570050-1025, or equivalent) with calibration verified. Ground glass stopper (size 19/22 joint) is used to prevent evaporation of extracts.

5.6.2 Evaporation flask--500 mL (Kontes K-570001-0500, or equivalent), attached to concentrator tube with springs (Kontes K-662750-0012).

5.6.3 Snyder column--three ball macro (Kontes K-503000-0232, or equivalent).

5.6.4 Snyder column--two ball micro (Kontes K-469002-0219, or equivalent).

5.6.5 Boiling chips--approx 10/40 mesh, extracted with methylene chloride and baked at 450 °C for one hr minimum.

5.7 Water bath--heated, with concentric ring cover, capable of temperature control (+/- 2 °C), installed in a fume hood.

5.8 Sample vials--amber glass, 2 - 5 mL with Teflon-lined screw cap.

5.9 Balances

5.9.1 Analytical--capable of weighing 0.1 mg.

5.9.2 Top loading--capable of weighing 10 mg.

5.10 Automated gel permeation chromatograph (Analytical Biochemical Labs, Inc, Model GPC Autoprep 1002, or equivalent)

5.10.1 Column--600 - 700 mm x 25 mm i.d., packed with 70 g of SX-3 Bio-beads

5.11 Gas chromatograph--shall have splitless or on-column injection port for capillary column, temperature program with 30 °C hold, and shall meet all of the performance specifications in section 12.

5.11.1 Column--30 +/-5 m x 0.25 +/- 0.02 mm i.d. 5% phenyl, 94% methyl, 1% vinyl silicone bonded phase fused silica capillary column (J & W DB-5, or equivalent).

5.12 Mass spectrometer--70 eV electron impact ionization, shall repetitively scan from 35 to 450 amu in 0.95 - 1.00 second, and shall produce a unit resolution (valleys between m/z 441-442 less than 10 percent of the height of the 441 peak), background corrected mass spectrum from 50 ng decafluorotriphenylphosphine (DFTPP) introduced through the GC inlet. The spectrum shall meet the mass-intensity criteria in table 7 (reference 6). The mass spectrometer shall be interfaced to the GC such that the end of the capillary column terminates within one centimeter of the ion source but does not intercept the electron or ion beams. All portions of the column which connect the GC to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds.

5.13 Data system--shall collect and record MS data, store mass-intensity data in spectral libraries, process GCMS data, generate reports, and shall compute and record response factors.

5.13.1 Data acquisition--mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device.

5.13.2 Mass spectral libraries--user created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GCMS runs for the compounds of interest (section 7.2).

5.13.3 Data processing--the data system shall be used to search, locate, identify, and quantify the compounds of interest in each GCMS analysis. Software routines shall be employed to compute retention times and peak areas. Displays of spectra, mass chromatograms, and library comparisons are required to verify results.

5.13.4 Response factors and multipoint calibrations--the data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and multi-point calibration curves (section 7). Computations of relative standard deviation (coefficient of variation) are used for testing calibration linearity. Statistics on initial (section 8.2) and on-going (section 12.7) performance shall be computed and maintained.

6 Reagents and standards

6.1 Reagents for adjusting sample pH

6.1.1 Sodium hydroxide--reagent grade, 6N in reagent water.

6.1.2 Sulfuric acid--reagent grade, 6N in reagent water.

6.2 Sodium sulfate--reagent grade, granular anhydrous, rinsed with methylene chloride (20 mL/g), baked at 450 °C for one hour minimum, cooled in a dessicator, and stored in a pre-cleaned glass bottle with screw cap which prevents moisture from entering.

6.3 Methylene chloride--distilled in glass (Burdick and Jackson, or equivalent).

6.4 GPC calibration solutions

6.4.1 Corn oil--200 mg/mL in methylene chloride

6.4.2 Bis(2-ethylhexyl)phthalate) and pentachlorophenol--4.0 mg/mL each in methylene chloride

6.5 Reference matrices

6.5.1 Reagent water--water in which the compounds of interest and interfering compounds are not detected by this method.

6.5.2 High solids reference matrix--playground sand or similar material in which the compounds of interest and interfering compounds are not detected by this method.

6.6 Standard solutions--purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96 percent or greater, the weight may be used without correction to compute the concentration of the standard. When not being used, standards are stored in the dark at -20 to -10 °C in screw-capped vials with Teflon-lined lids. A mark is placed on the vial at the level of the solution so that solvent evaporation loss can be detected. The vials are brought to room temperature prior to use. Any precipitate is redissolved and solvent is added if solvent loss has occurred.

6.7 Preparation of stock solutions--prepare in methylene chloride, benzene, p-dioxane, or a mixture of these solvents per the steps below. Observe the safety precautions in section 4. The large number of labeled and unlabeled acid and base/neutral compounds used for combined calibration (section 7) and calibration verification (12.5) require high concentrations (approx 40 mg/mL) when individual stock solutions are prepared, so that dilutions of mixtures will permit calibration with all compounds in a single set of solutions. The working range for most compounds is 10-200 ug/mL. Compounds with a reduced MS response may be prepared at higher concentrations.

6.7.1 Dissolve an appropriate amount of assayed reference material in a suitable solvent. For example, weigh 400 mg naphthalene in a 10 mL ground glass stoppered volumetric flask and fill to the mark with benzene. After the naphthalene is completely dissolved, transfer the solution to a 15 mL vial with Teflon-lined cap.

6.7.2 Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Qaulity control check samples that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

6.7.3 Stock standard solutions shall be replaced after six months, or sooner if comparison with quality control check standards indicates a change in concentration.

6.8 Labeled compound spiking solution--from stock standard solutions prepared as above, or from mixtures, prepare the spiking solution at a concentration of 200 ug/mL, or at a concentration appropriate to the MS response of each compound.

6.9 Secondary standard--using stock solutions (section 6.7), prepare a secondary standard containing all of the compounds in tables 1 and 2 at a concentration of 400 ug/mL, or higher concentration appropriate to the MS response of the compound.

6.10 Internal standard solution--prepare 2,2'-difluorobiphenyl (DFB) at a concentration of 10 mg/mL in benzene.

6.11 DFTPP solution--prepare at 50 ug/mL in acetone.

6.12 Solutions for obtaining authentic mass spectra (section 7.2)--prepare mixtures of compounds at concentrations which will assure authentic spectra are obtained for storage in libraries.

6.13 Calibration solutions--combine 0.5 mL of the solution in section 6.8 with 25, 50, 125, 250, and 500 uL of the solution in section 6.9 and bring to 1.00 mL total volume each. This will produce calibration solutions of nominal 10, 20, 50, 100 and 200 ug/mL of the pollutants and a constant nominal 100 ug/mL of the labeled compounds. Spike each solution with 10 uL of the internal standard solution (section 6.10). These solutions permit the relative response (labeled to unlabeled) to be measured as a function of concentration (section 7.4).

6.14 Precision and recovery standard--used for determination of initial (section 8.2) and on-going (section 12.7) precision and recovery. This solution shall contain the pollutants and labeled compounds at a nominal concentration of 100 ug/mL.

6.15 Stability of solutions--all standard solutions (sections 6.8 - 6.14) shall be analyzed within 48 hours of preparation and on a monthly basis thereafter for signs of degradation. Standards will remain acceptable if the peak area at the quantitation mass relative to the DFB internal standard remains within +/- 15 percent of the area obtained in the initial analysis of the standard.

7 Calibration

7.1 Assemble the GCMS and establish the operating conditions in table

5. Analyze standards per the procedure in section 11 to demonstrate that the analytical system meets the minimum levels in tables 5 and 6, and the mass-intensity criteria in table 7 for 50 ng DFTPP.

7.2 Mass spectral libraries--detection and identification of compounds of interest are dependent upon spectra stored in user created libraries.

7.2.1 Obtain a mass spectrum of each pollutant, labeled compound, and the internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components. That only a single compound is present is determined by examination of the spectrum. Fragments not attributable to the compound under study indicate the presence of an interfering compound.

7.2.2 Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum. An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak. Software algorithms designed to "enhance" the spectrum may eliminate distortion, but may also eliminate authentic masses or introduce other distortion.

7.2.3 The authentic reference spectrum is obtained under DFTPP tuning conditions (section 7.1 and table 7) to normalize it to spectra from other instruments.

7.2.4 The spectrum is edited by saving the 5 most intense mass spectral peaks and all other mass spectral peaks greater than 10 percent of the base peak. The spectrum may be further edited to remove common interfering masses. If 5 mass spectral peaks cannot be obtained under the scan conditions given in section 5.12, the mass spectrometer may be scanned to an m/z lower than 35 to gain additional spectral information. The spectrum obtained is stored for reverse search and for compound confirmation.

7.2.5 For the compounds in tables 3 and 4 and for other compounds for which the mass spectra, quantitation m/z's, and retention times are known but the instrument is not to be calibrated, add the retention time and reference compound (tables 5 and 6); the responsefactor and the quantitation m/z (tables 8 and 9); and spectrum (Appendix A) to the reverse search library. Edit the spectrum per section 7.2.4, if necessary.

7.3 Analytical range--demonstrate that 20 ng anthracene or phenanthrene produces an area at m/z 178 approx one-tenth that required to exceed the linear range of the system. The exact value must be determined by experience for each instrument. It is used to match the calibration range of the instrument to the analytical range and detection limits required, and to diagnose instrument sensitivity problems (section 15.3). The 20 ug/mL calibration standard (section 6.13) can be used to demonstrate this performance.

7.3.1 Polar compound detection--demonstrate that unlabeled pentachlorophenol and benzidine are detectable at the 50 ug/mL level (per all criteria in section 13). The 50 ug/mL calibration standard (section 6.13) can be used to demonstrate this performance.

7.4 Calibration with isotope dilution--isotope dilution is used when 1) labeled compounds are available, 2) interferences do not preclude its use, and 3) the quantitation m/z (tables 8 and 9) extracted ion current profile (EICP) area for the compound is in the calibration range. Alternate labeled compounds and quantitation m/z's may be used based on availability. If any of the above conditions preclude isotope dilution, the internal standard method (section 7.5) is used.

7.4.1 A calibration curve encompassing the concentration range is prepared for each compound to be determined. The relative response (pollutant to labeled) vs concentration in standard solutions is plotted or computed using a linear regression. The example in Figure 1 shows a calibration curve for phenol using phenol-d5 as the isotopic diluent. Also shown are the +/- 10 percent error limits (dotted lines). Relative Response (RR) is determined according to the procedures described below. A minimum of five data points are employed for calibration.

7.4.2 The relative response of a pollutant to its labeled analog is determined from isotope ratio values computed from acquired data. Three isotope ratios are used in this process:

R_x = the isotope ratio measured for the pure pollutant.

R_y = the isotope ratio measured for the labeled compound.

R_m = the isotope ratio of an analytical mixture of pollutant and labeled compounds.

The m/z's are selected such that $R_x > R_y$. If R_m is not between $2R_y$ and $0.5R_x$, the method does not apply and the sample is analyzed by the internal standard method.

7.4.3 Capillary columns usually separate the pollutant-labeled pair, with the labeled compound eluted first (figure 2). For this case,

R_x = [area m_1/z]/1, at the retention time of the pollutant (RT_2).

R_y = 1/[area m_2/z], at the retention time of the labeled compound RT_1)

R_m = [area at m_1/z (at RT_2)]/[area at m_2/z (at RT_1)], as measured in the mixture of the pollutant and labeled compounds (figure 2), and RR = R_m.

7.4.4 Special precautions are taken when the pollutant-labeled pair is not separated, or when another labeled compound with interfering spectral masses overlaps the pollutant (a case which can occur with isomeric compounds). In this case, it is necessary to determine the respective contributions of the pollutant and labeled compounds to the respective EICP areas. If the peaks are separated well enough to permit the data system or operator to remove the contributions of the compounds to each other, the equations in section 7.4.3 apply. This usually occurs when the height of the valley between the two GC peaks at the same m/z is less than 10 percent of the height of the shorter of the two peaks. If significant GC and spectral overlap occur, RR is computed using the following equation:

$RR = (R_y - R_m)(R_x + 1)/(R_m - R_x)(R_y + 1)$, where R_x is measured as shown in figure 3A, R_y is measured as shown in figure 3B, and R_m is measured as shown in figure 3C. For the example, $R_x = 46100/4780 = 9.644$, $R_y = 2650/43600 = 0.0608$, $R_m = 49200/48300 = 1.019$, and $RR = 1.114$.

7.4.5 To calibrate the analytical system by isotope dilution, analyze a 1.0 uL aliquot of each of the calibration standards (section 6.13) using the procedure in section 11. Compute the RR at each concentration.

7.4.6 Linearity--if the ratio of relative response to concentration for any compound is constant (less than 20 percent coefficient of variation) over the 5 point calibration range, an averaged relative response/concentration ratio may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range.

7.5 Calibration by internal standard--used when criteria for isotope dilution (section 7.4) cannot be met. The internal standard to be used for both acid and base/neutral analyses is 2,2'-difluorobiphenyl. The internal standard method is also applied to determination of compounds having no labeled analog, and to measurement of labeled compounds for intra-laboratory statistics (sections 8.4 and 12.7.4).

7.5.1 Response factors--calibration requires the determination of response factors (RF) which are defined by the following equation: $RF = (A_s \times C_{is})/(A_{is} \times C_s)$, where

A_s is the area of the characteristic mass for the compound in the daily standard

A_{is} is the area of the characteristric mass for the internal standard

C_{is} is the concentration of the internal standard (ug/mL)

C_s is the concentration of the compound in the daily standard (ug/mL)

7.5.1.1 The response factor is determined for at least five concentrations appropriate to the response of each compound (section 6.13); nominally, 10, 20, 50, 100, and 200 ug/mL. The amount of internal standard added to each extract is the same (100 ug/mL) so that C_{is} remains constant. The RF is plotted vs concentration for each compound in the standard (C_s) to produce a calibration curve.

7.5.1.2 Linearity--if the response factor (RF) for any compound is constant (less than 35 percent coefficient of variation) over the 5 point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point range.

7.6 Combined calibration--by using calibration solutions (section 6.13) containing the pollutants, labeled compounds, and the internal standard, a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods. These curves are verified each shift (section 12.5) by analyzing the 100 ug/mL calibration standard (section 6.13). Recalibration is required only if calibration verification (section 12.5) criteria cannot be met.

8 Quality assurance/quality control

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program (reference 7). The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

If the method is to be applied routinely to samples containing high solids with very little moisture (e.g., soils, filter cake, compost), the high solids reference matrix (section 6.5.2) is substituted for the reagent water (6.5.1) in all performance tests, and the high solids method (section 10) is used for these tests.

8.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in section 8.2.

8.1.2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met. Each time a modification is made to the method, the analyst is required to repeat the procedure in section 8.2 to demonstrate method performance.

8.1.3 Analyses of blanks are required to demonstrate freedom from contamination. The procedures and criteria for analysis of a blank are described in section 8.5.

8.1.4 The laboratory shall spike all samples with labeled compounds to monitor method performance. This test is described in section 8.3. When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits (section 15).

8.1.5 The laboratory shall, on an on-going basis, demonstrate through calibration verification and the analysis of the precision and recovery standard (section 6.14) that the analysis system is in control. These procedures are described in sections 12.1, 12.5, and 12.7.

8.1.6 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in section 8.4.

8.2 Initial precision and accuracy--to establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

8.2.1 For low solids (aqueous samples), extract, concentrate, and analyze two sets of four one-liter aliquots (8 aliquots total) of the precision and recovery standard (section 6.14) according to the procedure in section 10. For high solids samples, two sets of four 30 gram aliquots of the high solids reference matrix are used.

8.2.2 Using results of the first set of four analyses, compute the average recovery (X) in ug/mL and the standard deviation of the recovery (s) in ug/mL for each compound, by isotope dilution for pollutants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog.

8.2.3 For each compound, compare s and X with the corresponding limits for initial precision and accuracy in table 10. If s and X for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, system performance is unacceptable for that compound.

NOTE: The large number of compounds in table 10 present a substantial probability that one or more will fail the acceptance criteria when all compounds are analyzed. To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows:

8.2.4 Using the results of the second set of four analyses, compute s and X for only those compounds which failed the test of the first set of four analyses (section 8.2.3). If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin. If, however, any of the same compounds fail again, the analysis system is not performing properly for these compounds. In this event, correct the problem and repeat the entire test (section 8.2.1).

8.3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix.

8.3.1 Analyze each sample according to the method beginning in section 10.

8.3.2 Compute the percent recovery (P) of the labeled compounds using the internal standard method (section 7.5).

8.3.3 Compare the labeled compound recovery for each compound with the corresponding limits in table 10. If the recovery of any compound falls outside its warning limit, method performance is unacceptable for that compound in that sample. Therefore, the sample is complex. Water samples are diluted, and smaller amounts of soils, sludges, and sediments are reanalyzed per section 15.

8.4 As part of the QA program for the laboratory, method accuracy for samples shall be assessed and records shall be maintained. After the analysis of five samples or a given matrix type (water, soil, sludge, sediment) for which the labeled compounds pass the tests in section 8.3, compute the average percent recovery (P) and the standard deviation of the percent recovery (s_p) for the labeled compounds only. Express the accuracy assessment as a percent recovery interval from P $-2s_p$ to P $+ 2s_p$ for each matrix. For example, if P = 90% and s_p = 10% for five analyses of compost, the accuracy interval is expressed as 70 - 110%. Update the accuracy assessment for each compound in each matrix on a regular basis (e.g. after each 5 - 10 new accuracy measurements).

8.5 Blanks--reagent water and high solids reference matrix blanks are analyzed to demonstrate freedom from contamination.

8.5.1 Extract and concentrate a one liter reagent water blank or a high solids reference matrix blank with each sample lot (samples started through the extraction process on the same 8 hr shift, to a maximum of 20 samples). Analyze the blank immediately after analysis of the precision and recovery standard (section 6.14) to demonstrate freedom from contamination.

8.5.2 If any of the compounds of interest (tables 1 thru 4) or any potentially interfering compound is found in an aqueous blank at greater than 10 ug/L, or in a high solids reference matrix blank at greater than 100 ug/kg (assuming a response factor of 1 relative to the internal standard for compounds not listed in tables 1 thru 4), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

8.6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The standards used for calibration (section 7), calibration verification (section 12.5), and for initial (section 8.2) and on-going (section 12.7) precision and recovery should be identical, so that the most precise results will be obtained. The GCMS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analyses of semi-volatiles by this method.

8.7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when the internal standard method is used.

9 Sample collection, preservation, and handling

9.1 Collect samples in glass containers following conventional sampling practices (reference 8). Aqueous samples which flow freely are collected in refrigerated bottles using automatic sampling equipment. Solid samples are collected as grab samples using wide mouth jars.

9.2 Maintain samples at 0 - 4 °C from the time of collection until extraction. If residual chlorine is present in aqueous samples, add 80 mg sodium thiosulfate per liter of water. EPA methods 330.4 and 330.5 may be used to measure residual chlorine (reference 9).

9.3 Begin sample extraction within seven days of collection, and analyze all extracts within 40 days of extraction.

10 Sample extraction, concentration, and cleanup--samples containing one percent solids or less are extracted directly using continuous liquid/liquid extraction techniques (section 10.2.1 and figure 4). Samples containing one to 30 percent solids are diluted to the one percent level with reagent water (section 10.2.2) and extracted using continuous liquid/liquid extraction techniques. Samples containing greater than 30 percent solids are extracted using ultrasonic techniques (section 10.2.5)

10.1 Determination of percent solids

10.1.1 Weigh 5 - 10 g of sample into a tared beaker.

10.1.2 Dry overnight (12 hours minimum) at 110 +/- 5 °C, and cool in a dessicator.

10.1.3 Determine percent solids as follows:

$$\%\ \text{solids} = \frac{\text{weight of dry sample}}{\text{weight of wet sample}} \times 100$$

10.2 Preparation of samples for extraction

10.2.1 Samples containing one percent solids or less--extract sample directly using continuous liquid/liquid extraction techniques.

10.2.1.1 Measure 1.00 +/- 0.01 liter of sample into a clean 1.5 - 2.0 liter beaker.

10.2.1.2 Dilute aliquot--for samples which are expected to be difficult to extract, concentrate, or clean-up, measure an additional 100.0 +/- 1.0 mL into a clean 1.5 - 2.0 liter beaker and dilute to a final volume of 1.00 +/- 0.1 liter with reagent water.

10.2.1.3 Spike 0.5 mL of the labeled compound spiking solution (section 6.8) into the sample aliquots. Proceed to preparation of the QC aliquots for low solids samples (section 10.2.3).

10.2.2 Samples containing one to 30 percent solids

10.2.2.1 Mix sample thoroughly.

10.2.2.2 Using the percent solids found in 10.1.3, determine the weight of sample required to produce one liter of solution containing one percent solids as follows:

$$\text{sample weight} = \frac{1000}{\%\ \text{solids}}\ \text{grams}$$

10.2.2.3 Place the weight determined in 10.2.2.2 in a clean 1.5 - 2.0 liter beaker. Discard all sticks, rocks, leaves and other foreign material prior to weighing.

10.2.2.4 Dilute aliquot--for samples which are expected to be difficult to extract, concentrate, or clean-up, weigh an amount of sample equal to one-tenth the amount determined in 10.2.2.2 into a second clean 1.5 - 2.0 liter beaker. When diluted to 1.0 liter, this dilute aliquot will contain 0.1 percent solids.

10.2.2.5 Bring the sample aliquot(s) above to 100 - 200 mL volume with reagent water.

10.2.2.6 Spike 0.5 mL of the labeled compound spiking solution (section 6.8) into each sample aliquot.

10.2.2.7 Using a clean metal spatula, break any solid portions of the sample into small pieces.

10.2.2.8 Place the 3/4 in. horn on the ultrasonic probe approx 1/2 in. below the surface of each sample aliquot and pulse at 50 percent for three minutes at full power. If necessary, remove the probe from the solution and break any large pieces using the metal spatula or a stirring rod and repeat the sonication. Clean the probe with methylene chloride:acetone (1:1) between samples to preclude cross-contamination.

10.2.2.9 Bring the sample volume to 1.0 +/- 0.1 liter with reagent water.

10.2.3 Preparation of QC aliquots for samples containing low solids

(<30 percent).

10.2.3.1 For each sample or sample lot (to a maximum of 20) to be extracted at the same time, place three 1.0 +/- 0.01 liter aliquots of reagent water in clean 1.5 - 2.0 liter beakers.

10.2.3.2 Spike 0.5 mL of the labeled compound spiking solution (section 6.8) into one reagent water aliquot. This aliquot will serve as the blank.

10.2.3.3 Spike 1.0 mL of the precision and recovery standard (section 6.14) into the two remaining reagent water aliquots.

10.2.4 Stir and equilibrate all sample and QC solutions for 1 - 2 hours. Extract the samples and QC aliquots per section 10.3.

10.2.5 Samples containing 30 percent solids or greater

10.2.5.1 Mix the sample thoroughly

10.2.5.2 Weigh 30 +/- 0.3 grams into a clean 400 - 500 mL beaker. Discard all sticks, rocks, leaves and other foreign material prior to weighing.

10.2.5.3 Dilute aliquot--for samples which are expected to be difficult to extract, concentrate, or clean-up, weigh 3 +/- 0.03 grams into a clean 400 - 500 mL beaker.

10.2.5.4 Spike 0.5 mL of the labeled compound spiking solution (section 6.8) into each sample aliquot.

10.2.5.5 QC aliquots--for each sample or sample lot (to a maximum of 20) to be extracted at the same time, place three 30 +/- 0.3 gram aliquots of the high solids reference matrix in clean 400 - 500 mL beakers.

10.2.5.6 Spike 0.5 mL of the labeled compound spiking solution (section 6.8) into one high solids reference matrix aliquot. This aliquot will serve as the blank.

10.2.5.7 Spike 1.0 mL of the precision and recovery standard (section

6.14) into the two remaining high solids reference matrix aliquots. Extract, concentrate, and clean up the high solids samples per sections 10.4 through 10.8.

10.3 Continuous extraction of low solids (aqueous) samples--place 100 - 150 mL methylene chloride in each continuous extractor and 200 - 300 mL in each distilling flask.

10.3.1 Pour the sample(s), blank, and standard aliquots into the extractors. Rinse the glass containers with 50 - 100 mL methylene chloride and add to the respective extractors. Include all solids in the extraction process.

10.3.2 Base/neutral extraction--adjust the pH of the waters in the extractors to 12 - 13 with 6N NaOH while monitoring with a pH meter. Begin the extraction by heating the flask until the methylene chloride is boiling. When properly adjusted, 1 - 2 drops of methylene chloride per second will fall from the condensor tip into the water. Test and adjust the pH of the waters during the first 1 - 2 hr and during the fifth to tenth hr of extraction. Extract for 24 - 48 hours.

10.3.3 Remove the distilling flask, estimate and record the volume of extract (to the nearest 100 mL), and pour the contents through a drying column containing 7 to 10 cm anhydrous sodium sulfate. Rinse the distilling flask with 30 - 50 mL of methylene chloride and pour through the drying column. Collect the solution in a 500 mL K-D evaporator flask equipped with a 10 mL concentrator tube. Seal, label as the base/neutral fraction, and concentrate per sections 10.5 to 10.6.

10.3.4 Acid extraction--adjust the pH of the waters in the extractors to 2 or less using 6N sulfuric acid. Charge clean distilling flasks with 300 - 400 mL of methylene chloride. Test and adjust the pH of the waters during the first 1 - 2 hr and during the fifth to tenth hr of extraction. Extract for 24 - 48 hours. Repeat section 10.3.3, except label as the acid fraction.

10.4 Ultrasonic extraction of high solids samples

10.4.1 Add 60 grams of anhydrous sodium sulfate the sample and QC aliquot(s) (section 10.2.5) and mix thoroughly.

10.4.2 Add 100 +/- 10 mL of acetone:methylene chloride (1:1) to the sample and mix thoroughly.

10.4.3 Place the 3/4 in. horn on the ultrasonic probe approx 1/2 in. below the surface of the solvent but above the solids layer and pulse at 50 percent for three minutes at full power. If necessary, remove the probe from the solution and break any large pieces using the metal spatula or a stirring rod and repeat the sonication.

10.4.4 Decant the extracts through Whatman 41 filter paper using glass funnels and collect in 500 - 1000 mL graduated cylinders.

10.4.5 Repeat the extraction steps (10.4.2 - 10.4.4) twice more for each sample and QC aliquot. On the final extraction, swirl the sample or QC aliquot, pour into its respective glass funnel, and rinse with acetone:methylene chloride. Record the total extract volume.

10.4.6 Pour each extract through a drying column containing 7 to 10 cm of anhydrous sodium sulfate. Rinse the graduated cylinder with 30 - 50 mL of methylene chloride and pour through the drying column. Collect each extract in a 500 mL K-D evaporator flask equipped with a 10 mL concentrator tube. Seal and label as the high solids semi-volatile fraction. Concentrate and clean up the samples and QC aliquots per sections 10.5 through 10.8.

10.5 Macro concentration--concentrate the extracts in separate 500 mL K-D flasks equipped with 10 mL concentrator tubes.

10.5.1 Add 1 to 2 clean boiling chips to the flask and attach a three-ball macro Snyder column. Prewet the column by adding approx one mL of methylene chloride through the top. Place the K-D apparatus in a hot water bath so that the entire lower rounded surface of the flask is bathed with steam. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid has reached an apparent volume of 1 mL, remove the K-D apparatus from the bath and allow the solvent to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lowers joint into the concentrator tube with 1 - 2 mL of methylene chloride. A 5 mL syringe is recommended for this operation.

10.5.2 For performance standards (sections 8.2 and 12.7) and for blanks (section 8.5), combine the acid and base/neutral extracts for each at this point. Do not combine the acid and base/neutral extracts for aqueous samples.

10.6 Micro-concentration--Add a clean boiling chip and attach a two ball micro Snyder column to the concentrator tube. Prewet the column by adding approx 0.5 mL methylene chloride through the top. Place the apparatus in the hot water bath. Adjust the vertical position and the water temperature as required to complete the concentration in 5 - 10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid reaches an apparent volume of approx 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes. Remove the micro Snyder column and rinse its lower joint into the concentrator tube with approx

0.2 mL of methylene chloride. Adjust the final volume to 5.0 mL if the extract is to be cleaned up by GPC, or to 1.0 mL if it has been cleaned up or does not require clean-up.

10.7 Transfer the concentrated extract to a clean screw-cap vial. Seal the vial with a Teflon-lined lid, and mark the level on the vial. Label with the sample number and fraction, and store in the dark at -20 to -10 °C until ready for analysis.

10.8 GPC setup and calibration

10.8.1 Column packing

10.8.1.1 Place 75 +/- 5 g of SX-3 Bio-beads in a 400 - 500 mL beaker.

10.8.1.2 Cover the beads and allow to swell overnight (12 hours minimum).

10.8.1.3 Transfer the swelled beads to the column and pump solvent through the column, from bottom to top, at 4.5 - 5.5 mL/min.

10.8.1.4 After purging the column with solvent for 1 - 2 hours, adjust the column head pressure to 7 - 10 psig, and purge for 4 - 5 hours to remove air from the column. Maintain a head pressure of 7 - 10 psig.

10.8.2 Column calibration

10.8.2.1 Load 5 mL of the corn oil solution into sample loop number one, and 5 mL of the phthalate/phenol solution into sample loop number two.

10.8.2.2 Inject the corn oil and collect approx 10 mL fractions at two minute intervals for 36 minutes.

10.8.2.3 Inject the phthalate/phenol mixture and collect 15 mL fractions for 60 minutes.

10.8.2.4 Determine the corn oil elution pattern gravimetrically (by evaporation of each fraction and weighing the residue), or by UV, IR, or other technique which will detect the corn oil.

10.8.2.5 Determine the phthalate/phenol elution pattern by GC, GCMS, UV, IR, or other technique which will detect these compounds.

10.8.2.6 Plot a normalized chromatogram of the corn oil and phthalate/phenol mixture.

10.8.2.7 Choose the "dump time" to allow >85 percent removal of the corn oil and >85 percent recovery of the phthalate.

10.8.2.8 Choose the "collect time" to extend at least 10 minutes past the elution of the phenol.

10.8.2.9 Verify the calibration with pentachlorophenol after every 20 extracts. Calibration is verified if the recovery of the pentachlorophenol is greater than 85 percent. If calibration is not verified, the system shall be recalibrated using the corn oil and phthalate/phenol mixture.

10.9 Extract cleanup

10.9.1 Filter the extract or load through the filter holder to remove particulates. Load the 5.0 mL extract onto the column. The maximum capacity of the column is 0.5 - 1.0 gram. If necessary, split the extract into multiple aliquots to prevent column overload.

10.9.2 Elute the extract using the calibration data determined in 10.8.2. Collect the eluate in a clean 400 - 500 mL beaker.

10.9.3 Concentrate the cleaned up extract per section 10.5.

10.9.4 Rinse the sample loading tube thoroughly with methylene chloride between extracts to prepare for the next sample.

10.9.5 If a particularly dirty extract is encountered, a 5.0 mL methylene chloride blank shall be run through the system to check for carry-over.

10.9.6 Reconcentrate the extract to one mL and transfer to a screw-cap vial per sections 10.6 and 10.7.

11 GCMS analysis

11.1 Establish the operating conditions given in tables 5 or 6 for analysis of the base/neutral or acid extracts, respectively. For analysis of combined extracts (section 10.5.2 and 10.9.6), use the operating conditions in table 5.

11.2 Bring the concentrated extract (section 10.7) or standard (sections 6.13 - 6.14) to room temperature and verify that any precipitate has redissolved. Verify the level on the extract (sections 6.6 and 10.7) and bring to the mark with solvent if required.

11.3 Add the internal standard solution (section 6.10) to the extract (use 1.0 uL of solution per 0.1 mL of extract) immediately prior to injection to minimize the possibility of loss by evaporation, adsorption, or reaction. Mix thoroughly.

11.4 Inject a volume of the standard solution or extract such that 100 ng of the internal standard will be injected, using on-column or splitless injection. For 1 mL extracts, this volume will be 1.0 uL. Start the GC column initial isothermal hold upon injection. Start MS data collection after the solvent peak elutes. Stop data collection after the benzo(ghi)perylene or pentachlorophenol peak elutes for the base/neutral (or semi-volatile) or acid fraction, respectively. Return the column to the initial temperature for analysis of the next sample.

12 System and laboratory performance

12.1 At the beginning of each 8 hr shift during which analyses are performed, GCMS system performance and calibration are verified for all pollutants and labeled compounds. For these tests, analysis of the 100 ug/mL calibration standard (section 6.13) shall be used to verify all performance criteria. Adjustment and/or recalibration (per section 7) shall be performed until all performance criteria are met. Only after all performance criteria are met may samples, blanks, and precision and recovery standards be analyzed.

12.2 DFTPP spectrum validity--inject 1 uL of the DFTPP solution (section 6.11) either separately or within a few seconds of injection of the standard (section 12.1) analyzed at the beginning of each shift. The criteria in table 7 shall be met.

12.3 Retention times--the absolute retention time of 2,2'-difluorobiphenyl shall be within the range of 1078 to 1248 seconds and the relative retention times of all pollutants and labeled compounds shall fall within the limits given in tables 5 and 6.

12.4 GC resolution--the valley height between anthracene and phenanthrene at m/z 178 (or the analogs at m/z 188) shall not exceed 10 percent of the taller of the two peaks.

12.5 Calibration verification--compute the concentration of each pollutant (tables 1 and 2) by isotope dilution (section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (section 7.5). Compute the concentration of the labeled compounds by the internal standard method. These concentrations are computed based on the calibration data determined in section 7.

12.5.1 For each pollutant and labeled compound being tested, compare the concentration with the calibration verification limit in table 10. If all compounds meet the acceptance criteria, calibration has been verified and analysis of blanks, samples, and precision and recovery standards may proceed. If, however, any compound fails, the measurement system is not performing properly for that compound. In this event, prepare a fresh calibration standard or correct the problem causing the failure and repeat the test (section

12.1), or recalibrate (section 7).

12.6 Multiple peaks--each compound injected shall give a single, distinct GC peak.

12.7 On-going precision and accuracy.

12.7.1 Analyze the extract of one of the pair of precision and recovery standards (section 10) prior to analysis of samples from the same lot.

12.7.2 Compute the concentration of each pollutant (tables 1 and 2) by isotope dilution (section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (section 7.5). Compute the concentration of the labeled compounds by the internal standard method.

12.7.3 For each pollutant and labeled compound, compare the concentration with the limits for on-going accuracy in table 10. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, any individual concentration falls outside of the range given, system performance is unacceptable for that compound.

NOTE: The large number of compounds in table 10 present a substantial probability that one or more will fail when all compounds are analyzed. To determine if the extraction/concentration system is out of control or if the failure is caused by probability, proceed as follows:

12.7.3.1 Analyze the second aliquot of the pair of precision and recovery standards (section 10).

12.7.3.2 Compute the concentration of only those pollutants or labeled compounds that failed the previous test (section 12.7.3). If these compounds now pass, the extraction/concentration processes are in control and analysis of blanks and samples may proceed. If, however, any of the same compounds fail again, the extraction/concentration processes are not being performed properly for these compounds. In this event, correct the problem, re-extract the sample lot (section 10) and repeat the on-going precision and recovery test (section 12.7).

12.7.4 Add results which pass the specifications in section 12.7.3 to initial and previous on-going data for each compound in each matrix. Update QC charts to form a graphic representation of continued laboratory performance (Figure 5). Develop a statement of laboratory accuracy for each pollutant and labeled compound in each matrix type by calculating the average percent recovery (R) and the standard deviation of percent recovery (s_r). Express the accuracy as a recovery interval from R - $2s_r$ to R + $2s_r$. For example, if R = 95% and s_r = 5%, the accuracy is 85 - 105%.

13 Qualitative determination--identification is accomplished by comparison of data from analysis of a sample or blank with data stored in the mass spectral libraries. For compounds for which the relative retention times and mass spectra are known, identification is confirmed per sections 13.1 and 13.2. For unidentified GC peaks, the spectrum is compared to spectra in the EPA/NIH mass spectral file per section 13.3.

13.1 Labeled compounds and pollutants having no labeled analog (tables 1 thru 4):

13.1.1 The signals for all characteristic m/z's stored in the spectral library (section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.1.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two (0.5 to 2 times) for all masses stored in the library.

13.1.3 For the compounds for which the system has been calibrated (tables 1 and 2), the retention time shall be within the windows specified in tables 5 and 6, or within +/- 15 scans or +/- 15 seconds (whichever is greater) for compounds for which no window is specified.

13.1.4 For the compounds for which the system has not been calibrated but the relative retention times and mass spectra are known (tables 3 and 4), the retention time relative to the 2,2'-difluorobiphenyl internal standard shall be within +/- 30 scans or +/- 30 seconds (whichever is greater) based on the nominal retention time specified in tables 5 and 6.

13.2 Pollutants having a labeled analog (tables 1 and 2):

13.2.1 The signals for all characteristic m/z's stored in the spectral library (section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.2.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library.

13.2.3 The relative retention time between the pollutant and its labeled analog shall be within the windows specified in tables 5 and 6.

13.3 Unidentified GC peaks

13.3.1 The signals for masses specific to a GC peak shall all maximize within +/- 1 scan.

13.3.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two with the masses stored in the EPA/NIH Mass Spectral File.

13.4 M/z's present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or background ions. If the experimental mass spectrum is contaminated, or if identification is ambiguous, an experienced spectrometrist (section 1.4) is to determine the presence or absence of the compound.

14 Quantitative determination

14.1 Isotope dilution--by adding a known amount of a labeled compound to every sample prior to extraction, correction for recovery of the pollutant can be made because the pollutant and its labeled analog exhibit the same effects upon extraction, concentration, and gas chromatography. Relative response (RR) values for sample mixtures are used in conjunction with calibration curves described in section 7.4 to determine concentrations directly, so long as labeled compound spiking levels are constant. For the phenol example given in figure 1 (section 7.4.1), RR would be equal to 1.114. For this RR value, the phenol calibration curve given in figure 1 indicates a concentration of 27 ug/mL in the sample extract (C_{ex}).

14.2 Internal standard--compute the concentration in the extract using the response factor determined from calibration data (section 7.5) and the following equation:

$$C_{ex} \text{ (ug/mL)} = (A_s \times C_{is})/(A_{is} \times RF)$$

where C_{ex} is the concentration of the compound in the extract, and the other terms are as defined in section 7.5.1.

14.3 The concentration of the pollutant in the solid phase of the sample is computed using the concentration of the pollutant in the extract and the weight of the solids (section 10), as follows:

Concentration in solid (ug/kg) = $(C_{ex} \times V_{ex})/W_s$

where C_{ex} is the extract volume in mL and W_s is the sample weight in kg.

14.4 If the EICP area at the quantitation m/z for any compound exceeds the calibration range of the system, the extract of the dilute aliquot (section 10) is analyzed by isotope dilution; otherwise, the extract is diluted by a factor of 10, 9 uL of internal standard solution (section 6.10) are added to a 1.0 mL aliquot, and this diluted extract is analyzed by the internal standard method (section 14.2).

14.5 For GC peaks which are to be identified (per section 13.3), the sample is diluted by successive factors of 10 when any peak in the uncorrected mass spectrum at the GC peak maximum is saturated.

14.6 Results are reported for all pollutants, labeled compounds, and tentatively identified compounds found in all standards, blanks, and samples, in units of ug/L for aqueous samples or in ug/kg dry weight of solids for high solids samples (soils, sediments, filter cake, compost), to three significant figures. Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation m/z is within the calibration range (section 14.4) or at which no m/z in the spectrum is saturated (section 14.5). For compounds having a labeled analog, results are reported at the least dilute level at which the area at the quantitation m/z is within the calibration range (section 14.4) and the labeled compound recovery is within the normal range for the method (section 15.4).

15 Analysis of complex samples

15.1 Some samples may contain high levels (>1000 ug/L) of the compounds of interest, interfering compounds, and/or polymeric materials. Some samples will not concentrate to one mL (section 10.6); others will overload the GC column and/or mass spectrometer.

15.2 Analyze the dilute aliquot (section 10) when the sample will not concentrate to 1.0 mL. If a dilute aliquot was not extracted, and the sample holding time (section 9.3) has not been exceeded, dilute an aliquot of an aqueous sample with reagent water, or weigh a dilute aliquot of a high solids sample and re-extract (section 10); otherwise, dilute the extract (section 14.4) and analyze by the internal standard method (section 14.2).

15.3 Recovery of internal standard--the EICP area of the internal standard should be within a factor of two of the area in the shift standard (section 12.1). If the absolute areas of the labeled compounds are within a factor of two of the respective areas in the shift standard, and the internal standard area is less than one-half of its respective area, then internal standard loss in the extract has occurred. In this case, use one of the labeled compounds (preferably a polynuclear aromatic hydrocarbon) to compute the concentration of a pollutant with no labeled analog.

15.4 Recovery of labeled compounds--in most samples, labeled compound recoveries will be similar to those from reagent water or from the high solids reference matrix (section 12.7). If the labeled compound recovery is outside the limits given in table 10, the extract from the dilute aliquot (section 10) is analyzed as in section 14.4. If the recoveries of all labeled compounds and the internal standard are low (per the criteria above), then a loss in instrument sensitivity is the most likely cause. In this case, the 100 ug/mL calibration standard (section 12.1) shall be analyzed and calibration verified (section 12.5). If a loss in sensitivity has occurred, the instrument shall be repaired, the performance specifications in section 12 shall be met, and the extract reanalyzed. If a loss in instrument sensitivity has not occurred, the method does not work on the sample being analyzed and the result may not be reported for regulatory compliance purposes.

16 Method performance

16.1 Interlaboratory performance for this method is detailed in reference 10. Reference mass spectra, retention times, and response factors are from references 11 and 12. Results of initial tests of this method on municipal sludge can be found in reference 13.

16.2 A chromatogram of the 100 ug/mL acid/base/neutral calibration standard (section 6.13) is shown in figure 6.

References

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Table 1

Base/Neutral Extractable Compounds Determined by Calibrated GCMS Using Isotope Dilution and Internal Standard Techniques

	Pollutant				Labeled Compound		
Compound	**Storet**	**CAS Registry**	**EPA-EGD**	**NPDES**	**Analog**	**CAS Registry**	**EPA-EGD**
acenaphthene	34205	83-32-9	001 B	001 B	d10	15067-20-2	201 B
acenaphthylene	34200	208-96-8	077 B	002 B	d8	93951-97-4	277 B
anthracene	34220	120-12-7	078 B	003 B	d10	1719-06-8	278 B
benzidine	39120	92-87-5	005 B	004 B	d8	92890-63-6	205 B
benzo(a)anthracene	34526	56-55-3	072 B	005 B	d12	1718-53-2	272 B
benzo(b)fluoranthene	34230	205-99-2	074 B	007 B	d12	93951-98-5	274 B
benzo(k)fluoranthene	34242	207-08-9	075 B	009 B	d12	93952-01-3	275 B
benzo(a)pyrene	34247	50-32-8	073 B	006 B	d12	63466-71-7	273 B
benzo(ghi)perylene	34521	191-24-2	079 B	008 B	d12	93951-66-7	279 B
biphenyl (Appendix C)	81513	92-52-4	512 B		d10	1486-01-7	612 B
bis(2-chloroethyl) ether	34273	111-44-4	018 B	011 B	d8	93952-02-4	218 B
bis(2-chloroethoxy)methane	34278	111-91-1	043 B	010 B	d8	93966-78-0	243 B
bis(2-chloroisopropyl) ether	34283	108-60-1	042 B	012 B	d12	93951-67-8	242 B
bis(2-ethylhexyl) phthalate	39100	117-81-7	[illegible]66 B	013 B	d4	93951-87-2	266 B
4-bromophenyl phenyl ether	34636	101-55-3	[illegible] B	014 B	d5	93951-83-8	241 B
butyl benzyl phthalate	34292	85-68-7	[illegible]	015 B	d4	93951-88-3	267 B
n-C10 (Appendix C)	77427	124-18-5	[illegible]		d22	16416-29-8	617 B
n-C12 (Appendix C)	77588	112-40-3	[illegible]		d26	16416-30-1	606 B
n-C14 (Appendix C)	77691	629-59-4	51[illegible]				618 B
n-C16 (Appendix C)	77757	544-76-3	519 B		d34	15716-08-2	619 B
n-C18 (Appendix C)	77804	593-45-3	520 B				620 B
n-C20 (Appendix C)	77830	112-95-8	521 B		d42	62369-67-9	621 B
n-C22 (Appendix C)	77859	629-97-0	522 B				622 B
n-C24 (Appendix C)	77886	646-31-1	523 B		d50	16416-32-3	623 B
n-C26 (Appendix C)	77901	630-01-3	524 B				624 B
n-C28 (Appendix C)	78116	630-02-4	525 B				625 B
n-C30 (Appendix C)	78117	638-68-6	526 B		d62	93952-07-9	626 B
carbazole (4c)	77571	86-74-8	528 B		d8	38537-24-5	628 B
2-chloronaphthalene	34581	91-58-7	020 B	016 B	d7	93951-84-9	220 B
4-chlorophenyl phenyl ether	34641	7005-72-3	040 B	017 B	d5	93951-85-0	240 B
chrysene	34320	218-01-9	076 B	018 B	d12	1719-03-5	276 B
p-cymene (Appendix C)	77356	99-87-6	513 B		d14	93952-03-5	613 B
dibenzo(a,h)anthracene	34556	53-70-3	082 B	019 B	d14	13250-98-1	282 B
dibenzofuran (Appendix C & 4c)	81302	132-64-9	505 B		d8	93952-04-6	605 B
dibenzothiophene (Synfuel)	77639	132-65-0	504 B		d8	33262-29-2	604 B
di-n-butyl phthalate	39110	84-74-2	068 B	026 B	d4	93952-11-5	268 B
1,2-dichlorobenzene	34536	95-50-1	025 B	020 B	d4	2199-69-1	225 B
1,3-dichlorobenzene	34566	541-73-1	026 B	021 B	d4	2199-70-4	226 B
1,4-dichlorobenzene	34571	106-46-7	027 B	022 B	d4	3855-82-1	227 B
3,3'-dichlorobenzidine	34631	91-94-1	028 B	023 B	d6	93951-91-8	228 B
diethyl phthalate	34336	84-66-2	070 B	024 B	d4	93952-12-6	270 B
2,4-dimethylphenol	34606	105-67-9	034 A	003 A	d3	93951-75-8	234 A
dimethyl phthalate	34341	131-11-3	071 B	025 B	d4	93951-89-4	271 B
2,4-dinitrotoluene	34611	121-14-2	035 B	027 B	d3	93951-68-9	235 B
2,6-dinitrotoluene	34626	606-20-2	036 B	028 B	d3	93951-90-7	236 B
di-n-octyl phthalate	34596	117-84-0	069 B	029 B	d4	93952-13-7	269 B
diphenylamine (Appendix C)	77579	122-39-4	507 B		d10	37055-51-9	607 B
diphenyl ether (Appendix C)	77587	101-84-8	508 B		d10	93952-05-7	608 B
1,2-diphenylhydrazine	34346	122-66-7	037 B	030 B	d10	93951-92-9	237 B
fluoranthene	34376	206-44-0	039 B	031 B	d10	93951-69-0	231 B
fluorene	34381	86-73-7	080 B	032 B	d10	81103-79-9	080 B

hexachlorobenzene	39700	118-74-1	009 B	033 B	13C6	93952-14-8	209 B
hexachlorobutadiene	34391	87-68-3	052 B	034 B	13C4	93951-70-3	252 B
hexachloroethane	34396	67-72-1	012 B	036 B	13C	93952-15-9	212 B
hexachlorocyclopentadiene	34386	77-47-4	053 B	035 B	13C4	93951-71-4	253 B
ideno(1,2,3-cd)pyrene	34403	193-39-5	083 B	037 B			
isophorone	34408	78-59-1	054 B	038 B	d8	93952-16-0	254 B
naphthalene	34696	91-20-3	055 B	039 B	d8	1146-65-2	255 B
beta-naphthylamine (Appendix C)	82553	91-59-8	502 B		d7	93951-94-1	602 B
nitrobenzene	34447	98-95-3	056 B	040 B	d5	4165-60-0	256 B
N-nitrosodimethylamine	34438	62-75-9	061 B	041 B	d6	17829-05-9	261 B
N-nitrosodi-n-proplyamine	34428	621-64-7	063 B	042 B	d14	93951-96-3	263 B
N-nitrosodiphenylamine	34433	86-30-6	062 B	043 B	d6	93951-95-2	262 B
phenanthrene	34461	85-01-8	081 B	044 B	d10	1517-22-2	281 B
phenol	34694	108-95-2	065 A	010 A	d5	4165-62-2	265 A
alpha-picoline (Synfuel)	77088	109-06-8	503 B		d7	93951-93-0	503 B
pyrene	34469	129-00-0	084 B	045 B	d10	1718-52-1	284 B
styrene (Appendix C)	77128	100-42-5	510 B		d5	5161-29-5	610 B
alpha-terpineol (Appendix C)	77493	98-55-5	509 B		d3	93952-06-8	609 B
1,2,3-trichlorobenzene (4c)	77613	87-61-6	529 B		d3	3907-98-0	629 B
1,2,4-trichlorobenzene	34551	120-82-1	008 B	046 B	d3	93952-16-0	208 B

Table 2

Acid Extractable Compounds Determined by Calibrated GCMS Using Isotope Dilution and Internal Standard Techniques

Compound	Storet	Pollutant			Labeled Compound		
		CAS Registry	EPA-EGD	NPDES	Analog	CAS Registry	EPA-EGD
4-chloro-3-methylphenol	34452	59-50-7	022 A	008 A	d2	93951-72-5	222 A
2-chlorophenol	34586	95-57-8	024 A	001 A	d4	93951-73-6	224 A
2,4-dichlorophenol	34601	120-83-2	031 A	002 A	d3	93951-74-7	231 A
2,4-dinitrophenol	34616	5 28-5	059 A	005 A	d3	93951-77-0	259 A
2-methyl-4,6-dinitrophenol	34657	534-52-1	060 A	004 A	d2	93951-76-9	260 A
2-nitrophenol	34591	88-75-5	057 A	006 A	d4	93951-75-1	257 A
4-nitrophenol	34646	100-02-7	058 A	007 A	d4	93951-79-2	258 A
pentachlorophenol	39032	87-86-5	064 A	009 A	13C6	85380-74-1	264 A
2,3,6-trichlorophenol (4c)	77688	933-75-5	530 A		d2	93951-81-6	630 A
2,4,5-trichlorophenol (4c)		95-95-4	531 A		d2	93951-82-7	631 A
2,4,6-trichlorophenol	34621	88-06-2	021 A	011 A	d2	93951-80-5	221 A

Table 3

Base/neutral Extractable Compounds to be Determined by Reverse Search and Quantitation using Known Retention Times, Response Factors, Reference Compound, and Mass Spectra

EGD No.	Compound	CAS Registry
555	acetophenone	98-86-2
556	4-aminobiphenyl	92-67-1
557	aniline	62-53-3
558	o-anisidine	90-04-0
559	aramite	140-57-9
560	benzanthrone	82-05-3
561	1,3-benzenediol (resorcinol)	108-46-3
562	benzenethiol	108-98-5
563	2,3-benzofluorene	243-17-4
564	benzyl alcohol	100-51-6
565	2-bromochlorobenzene	694-80-4
566	3-bromochlorobenzene	108-37-2
567	4-chloro-2-nitroaniline	89-63-4
568	5-chloro-o-toluidine	95-79-4
569	4-chloroaniline	106-47-8
570	3-chloronitrobenzene	121-73-3
571	o-cresol	95-48-7
572	crotoxyphos	7700-17-6
573	2,6-di-tert-butyl-p-benzoquinone	719-22-2
574	2,4-diaminotoluene	95-80-7
575	1,2-dibromo-3-chloropropane	96-12-8
576	2,6-dichloro-4-nitroaniline	99-30-9
577	1,3-dichloro-2-propanol	96-23-1
578	2,3-dichloroaniline	608-27-5
579	2,3-dichloronitrobenzene	3209-22-1
580	1,2:3,4-diepoxybutane	1464-53-5
581	3,3'-dimethoxybenzidine	119-90-4
582	dimethyl sulfone	67-71-0
583	p-dimethylaminoazobenzene	60-11-7
584	7,12-dimethylbenz(a)anthracene	57-97-6
585	N,N-dimethylformamide	68-12-2
586	3,6-dimethylphenanthrene	1576-67-6
587	1,3-dinitrobenzene	100-25-4
588	diphenyldisulfide	882-33-7
589	ethyl methanesulfonate	62-50-0
590	ethylenethiourea	96-45-7
591	ethynylestradiol 3-methyl ether	72-33-3
592	hexachloropropene	1888-71-7
593	2-isopropylnaphthalene	2027-17-0
594	isosafrole	120-58-1
595	longifolene	475-20-7
596	malachite green	569-64-2
597	methapyrilene	91-80-5
598	methyl methanesulfonate	66-27-3
599	2-methylbenzothioazole	120-75-2
900	3-methylcholanthrene	56-49-5
901	4,4'-methylenebis(2-chloroaniline)	101-14-4
902	4,5-methylenephenanthrene	203-64-5
903	1-methylfluorene	1730-37-6
904	2-methylnaphthalene	91-57-6

905	1-methylphenanthrene	832-69-9
906	2-(methylthio)benzothiazole	120-75-2
907	1,5-naphthalenediamine	2243-62-1
908	1,4-naphthoquinone	130-15-4
909	alpha-naphthylamine	134-32-7
910	5-nitro-o-toluidine	99-55-8
911	2-nitroaniline	88-77-4
912	3-nitroaniline	99-09-2
913	4-nitroaniline	100-01-6
914	4-nitrobiphenyl	92-93-3
915	N-nitrosodi-n-butylamine	924-16-3
916	N-nitrosodiethylamine	55-18-5
917	N-nitrosomethylethylamine	10595-95-6
918	N-nitrosomethylphenylamine	614-00-6
919	N-nitrosomorpholine	59-89-2
920	N-nitrosopiperidine	100-75-4
921	pentachlorobenzene	608-93-5
922	pentachloroethane	76-01-7
923	pentamethylbenzene	700-12-9
924	perylene	198-55-0
925	phenacetin	62-44-2
926	phenothiazine	92-84-2
927	1-phenylnaphthalene	605-02-7
928	2-phenylnaphthalene	612-94-2
929	pronamide	23950-58-5
930	pyridine	110-86-1
931	safrole	94-59-7
932	squalene	7683-64-9
933	1,2,4,5-tetrachlorobenzene	95-94-3
934	thianaphthene (2,3-benzothiophene)	95-15-8
935	thioacetamide	62-55-5
936	thioxanthone	492-22-8
937	o-toluidine	95-53-4
938	1,2,3-trimethoxybenzene	634-36-6
939	2,4,5-trimethylaniline	137-17-7
940	triphenylene	217-59-4
941	tripropyleneglycol methyl ether	20324-33-8
942	1,3,5-trithiane	291-21-4

Table 4

Acid Extractable Compounds to be Determined by Reverse Search and Quantitation using Known Retention Times, Response Factors, Reference Compound, and Mass Spectra

EGD No.	Compound	CAS Registry
943	benzoic acid	65-85-0
944	p-cresol	106-44-5
945	3,5-dibromo-4-hydroxybenzonitrile	1689-84-5
946	2,6-dichlorophenol	87-65-0
947	hexanoic acid	142-62-1
948	2,3,4,6-tetrachlorophenol	58-90-2

Table 5

Gas Chromatography of Base/neutral Extractable Compounds

		Retention time			Minimum Level (3)	Method Detection Limit (4)	
EGD No. (1)	Compound	Mean (sec)	EGD Ref	Relative (2)	(ug/mL)	low solids (ug/kg)	high solids (ug/kg)
164	2,2'-difluorobiphenyl (int std)	1163	164	1.000 - 1.000	10		
930	pyridine	378	164	0.325			
261	N-nitrosodimethylamine-d6 (5)	378	164	0.286 - 0.364	50		
361	N-nitrosodimethylamine (5)	385	261	1.006 - 1.028	50	16	27
585	N,N-dimethylformamide	407	164	0.350			
580	1,2:3,4-diepoxybutane	409	164	0.352			
603	alpha picoline-d7	417	164	0.326 - 0.393	50		
703	alpha picoline	426	603	1.006 - 1.028	50	25	87
917	N-nitrosomethylethylamine	451	164	0.338			
598	methyl methanesulfonate	511	164	0.439			
610	styrene-d5	546	164	0.450 - 0.488	10		
710	styrene	549	610	1.002 - 1.009	10	149*	17
916	N-nitrosodiethylamine	570	164	0.490			
577	1,3-dichloro-2-propanol	589	164	0.506			
589	ethyl methanesulfonate	637	164	0.548			
582	dimethyl sulfone	649	164	0.558			
562	benzenethiol	667	164	0.574			
922	pentachloroethane	680	164	0.585			
557	aniline	694	164	0.597			
613	p-cymene-d14	742	164	0.624 - 0.652	10		
713	p-cymene	755	613	1.008 - 1.023	10	426*	912*
265	phenol-d5	696	164	0.584 - 0.613	10		
365	phenol	700	265	0.995 - 1.010	10	2501*	757*
218	bis(2-chloroethyl) ether-d8	696	164	0.584 - 0.607	10		
318	bis(2-chloroethyl) ether	704	218	1.007 - 1.016	10	32	22
617	n-decane-d22	698	164	0.585 - 0.615	10		
717	n-decane	720	617	1.022 - 1.038	10	299*	1188*
226	1,3-dichlorobenzene-d4	722	164	0.605 - 0.636	10		
326	1,3-dichlorobenzene	724	226	0.998 - 1.008	10	46	26
227	1,4-dichlorobenzene-d4	737	164	0.601 - 0.666	10		
327	1,4-dichlorobenzene	740	227	0.997 - 1.009	10	35	20
225	1,2-dichlorobenzene-d4	758	164	0.632 - 0.667	10		
325	1,2-dichlorobenzene	760	225	0.995 - 1.008	10	63	16
935	thioacetamide	768	164	0.660			
564	benzyl alcohol	785	164	0.675			
242	bis(2-chloroisopropyl) ether-d12	788	164	0.664 - 0.691	10		
342	bis(2-chloroisopropyl) ether	799	242	1.010 - 1.016	10	24	39
571	o-cresol	814	164	0.700			
263	N-nitrosodi-n-propylamine-d14 (5)	817	164	0.689 - 0.716	20		
363	N-nitrosodi-n-propylamine (5)	830	263	1.008 - 1.023	20	46	47
555	acetophenone	818	164	0.703			
212	hexachloroethane-13C	819	164	0.690 - 0.717	10		
312	hexachloroethane	823	212	0.999 - 1.001	10	58	55
937	o-toluidine	830	164	0.714			
919	N-nitrosomorpholine	834	164	0.717			
575	1,2-dibromo-3-chloropropene	839	164	0.721			
256	nitrobenzene-d5	845	164	0.706 - 0.727	10		
356	nitrobenzene	849	256	1.002 - 1.007	10	39	28
566	3-bromochlorobenzene	854	164	0.734			

565	2-bromochlorobenzene	880	164	0.757			
941	tripropylene glycol methyl ether	881	164	0.758			
254	isophorone-d8	881	164	0.747 - 0.767	10		
354	isophorone	889	254	0.999 - 1.017	10	8	5
942	1,3,5-trithiane	889	164	0.764			
920	N-nitrosopiperidine	895	164	0.770			
234	2,4-dimethylphenol-d3	921	164	0.781 - 0.803	10		
334	2,4-dimethylphenol	924	234	0.999 - 1.003	10	26	13
243	bis(2-chloroethoxy) methane-d6 (5)	933	164	0.792 - 0.807	10		
343	bis(2-chloroethoxy) methane (5)	939	243	1.000 - 1.013	10	26	23
208	1,2,4-trichlorobenzene-d3	955	164	0.813 - 0.830	10		
308	1,2,4-trichlorobenzene	958	208	1.000 - 1.005	10	49	24
558	o-anisidine	962	164	0.827			
255	naphthalene-d8	963	164	0.819 - 0.836	10		
355	naphthalene	967	255	1.001 - 1.006	10	62	42
934	thianapthene	971	164	0.835			
609	alpha-terpineol-d3	973	164	0.829 - 0.844	10		
709	alpha-terpineol	975	609	0.998 - 1.008	10	nd	nd
606	n-dodecane-d26	953	164	0.730 - 0.908	10		
706	n-dodecane	981	606	0.986 - 1.051	10	860*	3885*
629	1,2,3-trichlorobenzene-d3 (5)	1000	164	0.852 - 0.868	10		
729	1,2,3-trichlorobenzene (5)	1003	629	1.000 - 1.005	10	260*	164*
252	hexachlorobutadiene-13C4	1005	164	0.856 - 0.871	10		
352	hexachlorobutadiene	1006	252	0.999 - 1.002	10	46	22
918	N-nitrosomethylphenylamine	1006	164	0.865			
592	hexachloropropene	1013	164	0.871			
569	4-chloroaniline	1016	164	0.874			
570	3-chloronitrobenzene	1018	164	0.875			
915	N-nitrosodi-n-butylamine	1063	164	0.914			
923	pentamethylbenzene	1083	164	0.931			
561	1,3-benzenediol	1088	164	0.936			
931	safrole	1090	164	0.937			
939	2,4,5-trimethylaniline	1091	164	0.938			
904	2-methylnaphthalene	1098	164	0.944			
599	2-methylbenzothiazole	1099	164	0.945			
568	5-chloro-o-toluidine	1101	164	0.947			
938	1,2,3-trimethoxybenzene	1128	164	0.970			
933	1,2,4,5-tetrachlorobenzene	1141	164	0.981			
253	hexachlorocyclopentadiene-13C4	1147	164	0.976 - 0.986	10		
353	hexachlorocyclopentadiene	1142	253	0.999 - 1.001	10	nd	nd
594	isosafrole (cis or trans)	1147	164	0.986			
594	isosafrole (cis or trans)	1190	164	1.023			
578	2,3-dichloroaniline	1160	164	0.997			
574	2,4-diaminotoluene	1187	164	1.021			
220	2-chloronaphthalene-d7	1185	164	1.014 - 1.024	10		
320	2-chloronaphthalene	1200	220	0.997 - 1.007	10	80	59
518	n-tetradecane	1203	164	1.034	10	256	3533
612	biphenyl-d10	1195	164	1.016 - 1.027	10		
712	biphenyl	1205	612	1.001 - 1.006	10	67	55
608	diphenyl ether-d10	1211	164	1.036 - 1.047	10		
708	diphenyl ether	1216	608	0.997 - 1.009	10	44	12
579	2,3-dichloronitrobenzene	1214	164	1.044			
911	2-nitroaniline	1218	164	1.047			
908	1,4-naphthoquinone	1224	164	1.052			
595	longifolene	1225	164	1.053			
277	acenaphthylene-d8	1265	164	1.080 - 1.095	10		
377	acenaphthylene	1247	277	1.000 - 1.004	10	57	18
593	2-isopropylnaphthalene	1254	164	1.078			

587	1,3-dinitrobenzene	1255	164	1.079			
576	2,6-dichloro-4-nitroaniline	1259	164	1.083			
271	dimethyl phthalate-d4	1269	164	1.083 - 1.102	10		
371	dimethyl phthalate	1273	271	0.998 - 1.005	10	62	21
573	2,6-di-t-butyl-p-benzoquinone	1273	164	1.095			
236	2,6-dinitrotoluene-d3	1283	164	1.090 - 1.112	10		
336	2,6-dinitrotoluene	1300	236	1.001 - 1.005	10	55	47
912	3-nitroaniline	1297	164	1.115			
201	acenaphthene-d10	1298	164	1.107 - 1.125	10		
301	acenaphthene	1304	201	0.999 - 1.009	10	64	55
605	dibenzofuran-d8	1331	164	1.134 - 1.155	10		
705	dibenzofuran	1335	605	0.998 - 1.007	10	77	210*
921	pentachlorobenzene	1340	164	1.152			
909	alpha-naphthylamine	1358	164	1.168			
235	2,4-dinitrotoluene-d3	1359	164	1.152 - 1.181	10		
335	2,4-dinitrotoluene	1364	235	1.000 - 1.002	10	65	209*
602	beta-naphthylamine-d7	1368	164	1.163 - 1.189	50		
702	beta-naphthylamine	1371	602	0.996 - 1.007	50	49	37
590	ethylenethiourea	1381	164	1.187			
280	fluorene-d10	1395	164	1.185 - 1.214	10		
380	fluorene	1401	281	0.999 - 1.008	10	69	61
240	4-chlorophenyl phenyl ether-d5	1406	164	1.194 - 1.223	10		
340	4-chlorophenyl phenyl ether	1409	240	0.990 - 1.015	10	73	59
270	diethyl phthalate-d4	1409	164	1.197 - 1.229	10		
370	diethyl phthalate	1414	270	0.996 - 1.006	10	52	16
906	2-(methylthio)benzothiazole	1415	164	1.217			
567	4-chloro-2-nitroaniline	1421	164	1.222			
910	5-nitro-o-toluidine	1422	164	1.223			
913	4-nitroaniline	1430	164	1.230			
619	n-hexadecane-d34	1447	164	1.010 - 1.478	10		
719	n-hexadecane	1469	619	1.013 - 1.020	10	116*	644*
237	1,2-diphenylhydrazine-d8	1433	164	1.216 - 1.248	20		
337	1,2-diphenylhydrazine (6)	1439	237	0.999 - 1.009	20	48	27
607	diphenylamine-d10	1437	164	1.213 - 1.249	20		
707	diphenylamine	1439	607	1.000 - 1.007	20	58	54
262	N-nitrosodiphenylamine-d6	1447	164	1.225 - 1.252	20		
362	N-nitrosodiphenylamine (7)	1464	262	1.000 - 1.002	20	55	36
241	4-bromophenyl phenyl ether-d5 (5)	1495	164	1.271 - 1.307	10		
341	4-bromophenyl phenyl ether (5)	1498	241	0.990 - 1.015	10	55	17
925	phenacetin	1512	164	1.300			
903	1-methylfluorene	1514	164	1.302			
209	hexachlorobenzene-13C6	1521	164	1.288 - 1.327	10		
309	hexachlorobenzene	1522	209	0.999 - 1.001	10	51	48
556	4-aminobiphenyl	1551	164	1.334			
929	pronamide	1578	164	1.357			
281	phenanthrene-d10	1578	164	1.334 - 1.380	10		
520	n-octadecane	1580	164	1.359	10	134*	844*
381	phenanthrene	1583	281	1.000 - 1.005	10	42	22
278	anthracene-d10	1588	164	1.342 - 1.388	10		
378	anthracene	1592	278	0.998 - 1.006	10	52	21
604	dibenzothiophene-d8	1559	164	1.314 - 1.361	10		
704	dibenzothiophene	1564	604	1.000 - 1.006	10	72	71
588	diphenyldisulfide	1623	164	1.396			
914	4-nitrobiphenyl	1639	164	1.409			
927	1-phenylnaphthalene	1643	164	1.413			
628	carbazole-d8 (5)	1645	164	1.388 - 1.439	20		
728	carbazole (5)	1650	628	1.000 - 1.006	20	47	24
621	n-eicosane-d42	1655	164	1.184 - 1.662	10		

721	n-eicosane	1677	621	1.010 - 1.021	10	83	229*
907	1,5-naphthalenediamine	1676	164	1.441			
902	4,5-methylenephenanthrene	1690	164	1.453			
905	1-methylphenanthrene	1697	164	1.439			
268	di-n-butyl phthalate-d4	1719	164	1.446 - 1.510	10		
368	di-n-butyl phthalate	1723	268	1.000 - 1.003	10	64	80
928	2-phenylnaphthalene	1733	164	1.490			
586	3,6-dimethylphenanthrene	1763	164	1.516			
597	methapyrilene	1781	164	1.531			
926	phenothiazine	1796	164	1.544			
239	fluoranthene-d10	1813	164	1.522 - 1.596	10		
339	fluoranthene	1817	239	1.000 - 1.004	10	54	22
572	crotoxyphos	1822	164	1.567			
936	thioxanthone	1836	164	1.579			
284	pyrene-d10	1844	164	1.523 - 1.644	10		
384	pyrene	1852	284	1.001 - 1.003	10	40	48
205	benzidine-d8	1854	164	1.549 - 1.632	50		
305	benzidine	1853	205	1.000 - 1.002	50	nd	nd
522	n-docosane	1889	164	1.624	10	432*	447*
559	aramite	1901	164	1.635			
559	aramite	1916	164	1.647			
583	p-dimethylaminoazobenzene	1922	164	1.653			
563	2,3-benzofluorene	1932	164	1.661			
623	n-tetracosane-d50	1997	164	1.671 - 1.764	10		
723	n-tetracosane	2025	612	1.012 - 1.015	10	--	--
932	squalene	2039	164	1.753			
267	butylbenzyl phthalate-d4 (5)	2058	164	1.715 - 1.824	10		
367	butylbenzyl phthalate (5)	2060	267	1.000 - 1.002	10	60	65
276	chrysene-d12	2081	164	1.743 - 1.837	10		
376	chrysene	2083	276	1.000 - 1.004	10	51	48
901	4,4'methylenebis(2-chloroaniline)	2083	164	1.791			
272	benzo(a)anthracene-d12	2082	164	1.735 - 1.846	10		
372	benzo(a)anthracene	2090	272	0.999 - 1.007	10	61	47
581	3,3'-dimethoxybenzidine	2090	164	1.797			
228	3,3'-dichlorobenzidine-d6	2088	164	1.744 - 1.848	50		
328	3,3'-dichlorobenzidine	2086	228	1.000 - 1.001	50	62	111
940	triphenylene	2088	164	1.795			
560	benzanthrone	2106	164	1.811			
266	bis(2-ethylhexyl) phthalate-d4	2123	164	1.771 - 1.880	10		
366	bis(2-ethylhexyl) phthalate	2124	266	1.000 - 1.002	10	553*	1310*
524	n-hexacosane	2147	164	1.846	10	609*	886*
591	ethynylestradiol 3-methyl ether	2209	164	1.899			
269	di-n-octyl phthalate-d4	2239	164	1.867 - 1.982	10		
369	di-n-octyl phthalate	2240	269	1.000 - 1.002	10	72	62
525	n-octacosane	2272	164	1.954	10	492*	1810*
584	7,12-dimethylbenz(a)anthracene	2284	164	1.964			
274	benzo(b)fluoranthene-d12	2281	164	1.902 - 2.025	10		
374	benzo(b)fluoranthene	2293	274	1.000 - 1.005	10	54	30
275	benzo(k)fluoranthene-d12	2287	164	1.906 - 2.033	10		
375	benzo(k)fluoranthene	2293	275	1.000 - 1.005	10	95	20
924	perylene	2349	164	2.020			
273	benzo(a)pyrene-d12	2351	164	1.954 - 2.088	10		
373	benzo(a)pyrene	2350	273	1.000 - 1.004	10	52	15
626	n-triacontane-d62	2384	164	1.972 - 2.127	10		
726	n-triacontane	2429	626	1.011 - 1.028	10	252*	658*
596	malachite green	2382	164	2.048			
900	3-methylcholanthrene	2439	164	2.097			
083	indeno(1,2,3-cd)pyrene	2650	164	2.279	20	67	263*

282	dibenzo(a,h)anthracene-d14 (5)	2649	164	2.107 - 2.445	20		
382	dibenzo(a,h)anthracene (5)	2660	282	1.000 - 1.007	20	49	125
279	benzo(ghi)perylene-d12	2741	164	2.187 - 2.524	20		
379	benzo(ghi)perylene	2730	279	1.001 - 1.006	20	44	nd

(1) Reference numbers beginning with 0, 1, 5, or 9 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.
(2) Single values in this column are based on single laboratory data.
(3) This is a minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points. The concentration in the aqueous or solid phase is determined using the equations in section 14.
(4) Method detection limits determined in digested sludge (low solids) and in filter cake or compost (high solids).
(5) Specification derived from related compound.
(6) Detected as azobenzene
(7) Detected as diphenylamine
nd = not detected when spiked into the sludge tested
*Background levels of these compounds were present in the sludge tested, resulting in higher than expected MDL's. The MDL for these compounds is expected to be approximately 50 ug/kg with no interferences present.

Column: 30 +/- 2 m x 0.25 +/- 0.02 mm i.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary
Temperature program: 5 min at 30 °C; 30 - 280 °C at 8 °C per min; isothermal at 280 °C until benzo(ghi)perylene elutes
Gas velocity: 30 +/- 5 cm/sec at 30 °C

Table 6

Gas Chromatography of Acid Extractable Compounds

EGD No. (1)	Compound	Retention time Mean (sec)	Retention time EGD Ref	Retention time Relative (2)	Minimum Level (3) (ug/mL)	Method Detection Limit (4) low solids (ug/kg)	Method Detection Limit (4) high solids (ug/kg)
164	2,2'-difluorobiphenyl (int std)	1163	164	1.000 - 1.000	10		
224	2-chlorophenol-d4	701	164	0.587 - 0.618	10		
324	2-chlorophenol	705	224	0.997 - 1.010	10	18	10
947	hexanoic acid	746	164	0.641			
944	p-cresol	834	164	0.717			
257	2-nitrophenol-d4	898	164	0.761 - 0.783	20		
357	2-nitrophenol	900	257	0.994 - 1.009	20	39	44
231	2,4-dichlorophenol-d3	944	164	0.802 - 0.822	10		
331	2,4-dichlorophenol	947	231	0.997 - 1.006	10	24	116
943	benzoic acid	971	164	0.835			
946	2,6-dichlorophenol	981	164	0.844			
222	4-chloro-3-methylphenol-d2	1086	164	0.930 - 0.943	10		
322	4-chloro-3-methylphenol	1091	222	0.998 - 1.003	10	41	62
221	2,4,6-trichlorophenol-d2	1162	164	0.994 - 1.005	10	46	111
321	2,4,6-trichlorophenol	1165	221	0.998 - 1.004	10		
631	2,4,5-trichlorophenol-d2 (5)	1167	164	0.998 - 1.009	10		
731	2,4,5-trichlorophenol	1170	631	0.998 - 1.004	10	32	55
530	2,3,6-trichlorophenol	1195	164	1.028	10	58	37
259	2,4-dinitrophenol-d3	1323	164	1.127 - 1.149	50		
359	2,4-dinitrophenol	1325	259	1.000 - 1.005	50	565	642
258	4-nitrophenol-d4	1349	164	1.147 - 1.175	50		
358	4-nitrophenol	1354	258	0.997 - 1.006	50	287	11
948	2,3,4,6-tetrachlorophenol	1371	164	1.179			
260	2-methyl-4,6-dinitrophenol-d2	1433	164	1.216 - 1.249	20		
360	2-methyl-4,6-dinitrophenol	1435	260	1.000 - 1.002	20	385	83
945	3,5-dibromo-4-hydroxybenzonitrile	1481	164	1.273			
264	pentachlorophenol-13C6	1559	164	1.320 - 1.363	50		
364	pentachlorophenol	1561	264	0.998 - 1.002	50	51	207

(1) Reference numbers beginning with 0, 1, 5, or 9 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.
(2) Single values in this column are based on single laboratory data
(3) This is a minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points. The concentration in the aqueous or solid phase is determined using the equations in section 14.
(4) Method detection limits determined in digested sludge (low solids) and in filter cake or compost (high solids).
*Background levels of these compounds were present in the sludge resulting in higher than expected MDL's. The MDL for these compounds is expected to be approximately 50 ug/kg with no interferences present.
(5) Specification derived from related compound.

Column: 30 +/- 2 m x 0.25 +/- 0.02 mm i.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary
Temperature program: 5 min at 30 ^{o}C; 30 - 250 ^{o}C or until pentachlorophenol elutes

Gas velocity: 30 +/- 5 cm/sec at 30 °C

Table 7

DFTPP Mass-intensity Specifications*

Mass	Intensity required
51	8 - 82 percent of m/z 198
68	less than 2 percent of m/z 69
69	11 - 91 percent of m/z 198
70	less than 2 percent of m/z 69
127	32 - 59 percent of m/z 198
197	less than 1 percent of m/z 198
198	base peak, 100 percent abundance
199	4 - 9 percent of m/z 198
275	11 - 30 percent of m/z 198
441	44 - 110 percent of m/z 442
442	30 - 86 percent of m/z 198
443	14 - 24 percent of m/z 442

*Reference 6

Table 8

Base/neutral Extractable Compound Characteristic m/z's and Response Factors

Compound	Labeled analog	Primary m/z	Response Factor (1)
acenaphthene	d10	154/164	
acenaphthylene	d8	152/160	
acetophenone		105	0.79
4-aminobiphenyl		169	0.81
aniline		93	1.04
o-anisidine		108	0.43
anthracene	d10	178/188	
aramite		185	0.19
benzanthrone		230	0.15
1,3-benzenediol		110	0.78
benzenethiol		110	0.18
benzidine	d8	184/192	
benzo(a)anthracene	d12	228/240	
benzo(b)fluoranthene	d12	252/264	
benzo(k)fluoranthene	d12	252/264	
benzo(a)pyrene	d12	252/264	
benzo(ghi)perylene	d12	276/288	
2,3-benzofluorene		216	0.35
benzoic acid		105	0.16
benzyl alcohol		79	0.47
biphenyl	d10	154/164	
bis(2-chloroethyl) ether	d8	93/101	
bis(2-chloroethoxy)methane	d8	93/97	
bis(2-chloroisopropyl) ether	d12	121/131	
bis(2-ethylhexyl) phthalate	d4	149/153	
2-bromochlorobenzene		111	0.33
3-bromochlorobenzene		192	0.40
4-bromophenyl phenyl ether	d5	248/255	
butyl benzyl phthalate	d4	149/153	
n-C10	d22	55/66	
n-C12	d26	55/66	
n-C14		55	
n-C16	d34	55/66	
n-C18		55	
n-C20	d42	55/66	
n-C22		55	
n-C24	d50	55/66	
n-C26		55	
n-C28		55	
n-C30	d62	55/66	
carbazole	d8	167/175	
4-chloro-2-nitroaniline		172	0.20
5-chloro-o-toluidine		106	0.50
4-chloroaniline		127	0.73
2-chloronaphthalene	d7	162/169	
3-chloronitrobenzene		157	0.18
4-chlorophenyl phenyl ether	d5	204/209	
3-chloropropionitrile		54	0.42
chrysene	d12	228/240	
o-cresol		108	0.59
crotoxyphos		127	0.017

p-cymene	d14	119/130	
2,6-di-tert-butyl-p-benzoquinone		220	0.078
di-n-butyl phthalate	d4	149/153	
2,4-diaminotoluene		122	0.059
dibenzo(a,h)anthracene	d14	278/292	
dibenzofuran	d8	168/176	
dibenzothiophene	d8	184/192	
1,2-dibromo-3-chloropropane		157	0.22
2,6-dichloro-4-nitroaniline		124	0.019
1,3-dichloro-2-propanol		79	0.68
2,3-dichloroaniline		161	0.47
1,2-dichlorobenzene	d4	146/152	
1,3-dichlorobenzene	d4	146/152	
1,4-dichlorobenzene	d4	146/152	
3,3'-dichlorobenzidine	d6	252/258	
2,2'-difluorobiphenyl (int std)		190	
2,3-dichloronitrobenzene		191	0.11
1,2:3,4-diepoxybutane		55	0.27
diethyl phthalate	d4	149/153	
3,3'-dimethoxybenzidine		244	0.19
dimethyl phthalate	d4	163/167	
dimethyl sulfone		79	0.40
p-dimethylaminoazobenzene		120	0.23
7,12-dimethylbenz(a)anthracene		256	0.58
N,N-dimethylformamide		73	0.51
3,6-dimethylphenanthrene		206	0.72
2,4-dimethylphenol	d3	122/125	
1,3-dinitrobenzene		168	0.24
2,4-dinitrotoluene	d3	165/168	
2,6-dinitrotoluene	d3	165/167	
di-n-octyl phthalate	d4	149/153	
diphenylamine	d10	169/179	
diphenyl ether	d10	170/180	
diphenyldisulfide		218	0.25
1,2-diphenylhydrazine (2)	d10	77/82	
ethyl methanesulfonate		109	0.28
ethylenethiourea		102	0.22
ethynylestradiol 3-methyl ether		227	0.28
fluoranthene	d10	202/212	
fluorene	d10	166/176	
hexachlorobenzene	^{13}C6	284/292	
hexachlorobutadiene	^{13}C4	225/231	
hexachloroethane	^{13}C	201/204	
hexachlorocyclopentadiene	^{13}C4	237/241	
hexachloropropene		213	0.23
indeno(1,2,3-cd)pyrene		276	
isophorone	d8	82/88	
2-isopropylnaphthalene		170	0.32
isosafrole		162	0.33
longifolene		161	0.14
malachite green		330	
methapyrilene		97	0.43
methyl methanesulfonate		80	0.20
2-methylbenzothiazole		149	0.59
3-methylcholanthrene		268	0.59
4,4'-methylenebis(2-chloroaniline)		231	0.21
4,5-methylenephenanthrene		190	0.44
1-methylfluorene		180	0.37

2-methylnaphthalene		142	0.99
1-methylphenanthrene		192	0.65
2-(methylthio)benzothiazole		181	0.42
naphthalene	d8	128/136	
1,5-naphthalenediamine		158	0.085
1,4-naphthoquinone		158	0.021
alpha-naphthylamine		143	0.89
beta-naphthylamine	d7	143/150	
5-nitro-o-toluidine		152	0.31
2-nitroaniline		138	0.39
3-nitroaniline		138	0.27
4-nitroaniline		138	0.11
nitrobenzene	d5	128/128	
4-nitrobiphenyl		199	0.35
N-nitrosodi-n-butylamine		84	0.47
N-nitrosodi-n-propylamine	d14	70/78	
N-nitrosodiethylamine		102	0.45
N-nitrosodimethylamine	d6	74/80	
N-nitrosodiphenylamine (3)	d6	169/175	
N-nitrosomethylethylamine		88	0.33
N-nitrosomethylphenylamine		106	0.024
N-nitrosomorpholine		56	0.49
N-nitrosopiperidine		114	0.41
pentachlorobenzene		248	0.25
pentachloroethane		117	0.20
pentamethylbenzene		148	0.42
perylene		252	0.30
phenacetin		108	0.38
phenanthrene	d10	178/188	
phenol	d5	94/71	
phenothiazine		199	0.15
1-phenylnaphthalene		204	0.48
2-phenylnaphthalene		204	0.73
alpha-picoline	d7	93/100	
pronamide		173	0.31
pyrene	d10	202/212	
pyridine		79	0.68
safrole		162	0.45
squalene		69	0.042
styrene	d5	104/109	
alpha-terpineol	d3	59/62	
1,2,4,5-tetrachlorobenzene		216	0.43
thianaphthene		134	1.52
thioacetamide		75	0.28
thioxanthone		212	0.23
o-toluidine		106	1.04
1,2,3-trichlorobenzene	d3	180/183	
1,2,4-trichlorobenzene	d3	180/183	
1,2,3-trimethoxybenzene		168	0.48
2,4,5-trimethylaniline		120	0.28
triphenylene		228	1.32
tripropylene glycol methyl ether		59	0.092
1,3,5-trithiane		138	0.15

(1) referenced to 2,2'-difluorobiphenyl
(2) detected as azobenzene
(3) detected as diphenylamine

Table 9

Acid Extractable Compound Characteristic m/z's

Compound	Labeled analog	Primary m/z	Response Factor (1)
benzoic acid		105	0.16
4-chloro-3-methylphenol	d2	107/109	
2-chlorophenol	d4	128/132	
p-cresol		108	0.61
3,5-dibromo-4-hydroxybenzonitrile		277	0.12
2,4-dichlorophenol	d3	162/167	
2,6-dichlorophenol		162	0.42
2,4-dinitrophenol	d3	184/187	
hexanoic acid		60	0.62
2-methyl-4,6-dinitrophenol	d2	198/200	
2-nitrophenol	d4	139/143	
4-nitrophenol	d4	139/143	
pentachlorophenol	^{13}C6	266/272	
2,3,4,6-tetrachlorophenol		232	0.17
2,3,6-trichlorophenol	d2	196/200	
2,4,5-trichlorophenol	d2	196/200	
2,4,6-trichlorophenol	d2	196/200	

(1) referenced to 2,2'-difluorobiphenyl

Table 10

Acceptance Criteria for Performance Tests

		Acceptance criteria				
EGD No. (1)	Compound	Initial precision and accuracy Section 8.2.3 (ug/L)		labeled compound recovery Sec 8.3 and 14.2	calibration verification Sec 12.5	On-going accuracy Sec 12.7
		s	X	P (%)	(ug/mL)	R (ug/L)
301	acenaphthene	21	79 - 134		80 - 125	72 - 144
201	acenaphthene-d10	38	38 - 147	20 - 270	71 - 141	30 - 180
377	acenaphthylene	38	69 - 186		60 - 166	61 - 207
277	acenaphthylene-d8	31	39 - 146	23 - 239	66 - 152	33 - 168
378	anthracene	41	58 - 174		60 - 168	50 - 199
278	anthracene-d10	49	31 - 194	14 - 419	58 - 171	23 - 242
305	benzidine	119	16 - 518		34 - 296	11 - 672
205	benzidine-d8	269	ns(2) ns	ns - ns	ns - ns	ns - ns
372	benzo(a)anthracene	20	65 - 168		70 - 142	62 - 176
272	benzo(a)anthracene-d12	41	25 - 298	12 - 605	28 - 357	22 - 329
374	benzo(b)fluoranthene	183	32 - 545		61 - 164	20 - ns
274	benzo(b)fluoranthene-d12	168	11 - 577	ns - ns	14 - ns	ns - ns
375	benzo(k)fluoranthene	26	59 - 143		13 - ns	53 - 155
275	benzo(k)fluoranthene-d12	114	15 - 514	ns - ns	13 - ns	ns - 685
373	benzo(a)pyrene	26	62 - 195		78 - 129	59 - 206
273	benzo(a)pyrene-d12	24	35 - 181	21 - 290	12 - ns	32 - 194
379	benzo(ghi)perylene	21	72 - 160		69 - 145	58 - 168
279	benzo(ghi)perylene-d12	45	29 - 268	14 - 529	13 - ns	25 - 303
712	biphenyl (Appendix C)	41	75 - 148		58 - 171	62 - 176
612	biphenyl-d10	43	28 - 165	ns - ns	52 - 192	17 - 267
318	bis(2-chloroethyl) ether	34	55 - 196		61 - 164	50 - 213
218	bis(2-chloroethyl) ether-d8	33	29 - 196	15 - 372	52 - 194	25 - 222
343	bis(2-chloroethoxy)methane	27	43 - 153		44 - 228	39 - 166
243	bis(2-chloroethoxy)methane-d8(3)	33	29 - 196	15 - 372	52 - 194	25 - 222
342	bis(2-chloroisopropyl) ether	17	81 - 138		67 - 148	77 - 145
242	bis(2-chloroisopropyl)ether-d12	27	35 - 149	20 - 260	44 - 229	30 - 169
366	bis(2-ethylhexyl) phthalate	31	69 - 220		76 - 131	64 - 232
266	bis(2-ethylhexyl) phthalate-d4	29	32 - 205	18 - 364	43 - 232	28 - 224
341	4-bromophenyl phenyl ether	44	44 - 140		52 - 193	35 - 172
241	4-bromophenylphenyl ether-d5(3)	52	40 - 161	19 - 325	57 - 175	29 - 212
367	butyl benzyl phthalate	31	19 - 233		22 - 450	35 - 170
267	butyl benzyl phthalate-d4 (3)	29	32 - 205	18 - 364	43 - 232	28 - 224
717	n-C10 (Appendix C)	51	24 - 195		42 - 235	19 - 237
617	n-C10-d22	70	ns - 298	ns - ns	44 - 227	ns - 504
706	n-C12 (Appendix C)	74	35 - 369		60 - 166	29 - 424
606	n-C12-d26	53	ns - 331	ns - ns	41 - 242	ns - 408
518	n-C14 (Appendix C) (3)	109	ns - ns		37 - 268	ns - ns
719	n-C16 (Appendix C)	33	80 - 162		72 - 138	71 - 131
619	n-C16-d34	46	37 - 162	18 - 308	54 - 186	28 - 202
520	n-C18 (Appendix C) (3)	39	42 - 131		40 - 249	35 - 167
721	n-C20 (Appendix C)	59	53 - 263		54 - 184	46 - 301
621	n-C20-d42	34	34 - 172	19 - 306	62 - 162	29 - 198
522	n-C22 (Appendix C) (3)	31	45 - 152		40 - 249	39 - 195
723	n-C24 (Appendix C)	11	80 - 139		65 - 154	78 - 142
623	n-C24-d50	28	27 - 211	15 - 376	50 - 199	25 - 229
524	n-C26 (Appendix C) (3)	35	35 - 193		26 - 392	31 - 212
525	n-C28 (Appendix C) (3)	35	35 - 193		26 - 392	31 - 212

726	n-C30 (Appendix C)	32	61 - 200		66 - 152	56 - 215
626	n-C30-d62	41	27 - 242	13 - 479	24 - 423	23 - 274
728	carbazole (4c)	38	36 - 165		44 - 227	31 - 188
628	carbazole-d8 (3)	31	48 - 130	29 - 215	69 - 145	40 - 156
320	2-chloronaphthalene	100	46 - 357		58 - 171	35 - 442
220	2-chloronaphthalene-d7	41	30 - 168	15 - 324	72 - 139	24 - 204
322	4-chloro-3-methylphenol	37	76 - 131		85 - 115	62 - 159
222	4-chloro-3-methylphenol-d2	111	30 - 174	ns - 613	68 - 147	14 - 314
324	2-chlorophenol	13	79 - 135		78 - 129	76 - 138
224	2-chlorophenol-d4	24	36 - 162	23 - 255	55 - 180	33 - 176
340	4-chlorophenyl phenyl ether	42	75 - 166		71 - 142	63 - 194
240	4-chlorophenyl phenyl ether-d5	52	40 - 161	19 - 325	57 - 175	29 - 212
376	chrysene	51	59 - 186		70 - 142	48 - 221
276	chrysene-d12	69	33 - 219	13 - 512	24 - 411	23 - 290
713	p-cymene (Appendix C)	18	76 - 140		79 - 127	72 - 147
613	p-cymene-d14	67	ns - 359	ns - ns	66 - 152	ns - 468
382	dibenzo(a,h)anthracene	55	23 - 299		13 - 761	19 - 340
282	dibenzo(a,h)anthracene-d14 (3)	45	29 - 268	14 - 529	13 - ns	25 - 303
705	dibenzofuran (Appendix C)	20	85 - 136		73 - 136	79 - 146
605	dibenzofuran-d8	31	47 - 136	28 - 220	66 - 150	39 - 160
704	dibenzothiophene (Synfuel)	31	79 - 150		72 - 140	70 - 168
604	dibenzothiophene-d8	31	48 - 130	29 - 215	69 - 145	40 - 156
368	di-n-butyl phthalate	15	76 - 165		71 - 142	74 - 169
268	di-n-butyl phthalate-d4	23	23 - 195	13 - 346	52 - 192	22 - 209
325	1,2-dichlorobenzene	17	73 - 146		74 - 135	70 - 152
225	1,2-dichlorobenzene-d4	35	14 - 212	ns - 494	61 - 164	11 - 247
326	1,3-dichlorobenzene	43	63 - 201		65 - 154	55 - 225
226	1,3-dichlorobenzene-d4	48	13 - 203	ns - 550	52 - 192	ns - 260
327	1,4-dichlorobenzene	42	61 - 194		62 - 161	53 - 219
227	1,4-dichlorobenzene-d4	48	15 - 193	ns - 474	65 - 153	11 - 245
328	3,3'-dichlorobenzidine	26	68 - 174		77 - 130	64 - 185
228	3,3'-dichlorobenzidine-d6	80	ns - 562	ns - ns	18 - 558	ns - ns
331	2,4-dichlorophenol	12	85 - 131		67 - 149	83 - 135
231	2,4-dichlorophenol-d3	28	38 - 164	24 - 260	64 - 157	34 - 182
370	diethyl phthalate	44	75 - 196		74 - 135	65 - 222
270	diethyl phthalate-d4	78	ns - 260	ns - ns	47 - 211	ns - ns
334	2,4-dimethylphenol	13	62 - 153		67 - 150	60 - 156
234	2,4-dimethylphenol-d3	22	15 - 228	ns - 449	58 - 172	14 - 242
371	dimethyl phthalate	36	74 - 188		73 - 137	67 - 207
271	dimethyl phthalate-d4	108	ns - 640	ns - ns	50 - 201	ns - ns
359	2,4-dinitrophenol	18	72 - 134		75 - 133	68 - 141
259	2,4-dinitrophenol-d3	66	22 - 308	ns - ns	39 - 256	17 - 378
335	2,4-dinitrotoluene	18	75 - 158		79 - 127	72 - 164
235	2,4-dinitrotoluene-d3	37	22 - 245	10 - 514	53 - 187	19 - 275
336	2,6-dinitrotoluene	30	80 - 141		55 - 183	70 - 159
236	2,6-dinitrotoluene-d3	59	44 - 184	17 - 442	36 - 278	31 - 250
369	di-n-octyl phthalate	16	77 - 161		71 - 140	74 - 166
269	di-n-octyl phthalate-d4	46	12 - 383	ns - ns	21 - 467	10 - 433
707	diphenylamine (Appendix C)	45	58 - 205		57 - 176	51 - 231
607	diphenylamine-d10	42	27 - 206	11 - 488	59 - 169	21 - 249
708	diphenyl ether (Appendix C)	19	82 - 136		83 - 120	77 - 144
608	diphenyl ether-d10	37	36 - 155	19 - 281	77 - 129	29 - 186
337	1,2-diphenylhydrazine	73	49 - 308		75 - 134	40 - 360
237	1,2-diphenylhydrazine-d10	35	31 - 173	17 - 316	58 - 174	26 - 200
339	fluoranthene	33	71 - 177		67 - 149	64 - 194
239	fluoranthene-d10	35	36 - 161	20 - 278	47 - 215	30 - 187
380	fluorene	29	81 - 132		74 - 135	70 - 151
280	fluorene-d10	43	51 - 131	27 - 238	61 - 164	38 - 172

309	hexachlorobenzene	16	90 - 124		78 - 128	85 - 132
209	hexachlorobenzene-13C6	81	36 - 228	13 - 595	38 - 265	23 - 321
352	hexachlorobutadiene	56	51 - 251		74 - 135	43 - 287
252	hexachlorobutadiene-13C4	63	ns - 316	ns - ns	68 - 148	ns - 413
312	hexachloroethane	227	21 - ns		71 - 141	13 - ns
212	hexachloroethane-13C1	77	ns - 400	ns - ns	47 - 212	ns - 563
353	hexachlorocyclopentadiene	15	69 - 144		77 - 129	67 - 148
253	hexachlorocyclopentadiene-13C4	60	ns - ns	ns - ns	47 - 211	ns - ns
083	ideno(1,2,3-cd)pyrene (3)	55	23 - 299		13 - 761	19 - 340
354	isophorone	25	76 - 156		70 - 142	70 - 168
254	isophorone-d8	23	49 - 133	33 - 193	52 - 194	44 - 147
360	2-methyl-4,6-dinitrophenol	19	77 - 133		69 - 145	72 - 142
260	2-methyl-4,6-dinitrophenol-d2	64	36 - 247	16 - 527	56 - 177	28 - 307
355	naphthalene	20	80 - 139		73 - 137	75 - 149
255	naphthalene-d8	39	28 - 157	14 - 305	71 - 141	22 - 192
702	beta-naphthylamine (Appendix C)	49	10 - ns		39 - 256	ns - ns
602	beta-naphthylamine-d7	33	ns - ns	ns - ns	44 - 230	ns - ns
356	nitrobenzene	25	69 - 161		85 - 115	65 - 169
256	nitrobenzene-d5	28	18 - 265	ns - ns	46 - 219	15 - 314
357	2-nitrophenol	15	78 - 140		77 - 129	75 - 145
257	2-nitrophenol-d4	23	41 - 145	27 - 217	61 - 163	37 - 158
358	4-nitrophenol	42	62 - 146		55 - 183	51 - 175
258	4-nitrophenol-d4	188	14 - 398	ns - ns	35 - 287	ns - ns
361	N-nitrosodimethylamine	49	10 - ns		39 - 256	ns - ns
261	N-nitrosodimethylamine-d6 (3)	33	ns - ns	ns - ns	44 - 230	ns - ns
363	N-nitrosodi-n-propylamine	45	65 - 142		68 - 148	53 - 173
263	N-nitrosodi-n-propylamine (3)	37	54 - 126	26 - 256	59 - 170	40 - 166
362	N-nitrosodiphenylamine	45	65 - 142		68 - 148	53 - 173
262	N-nitrosodiphenylamine-d6	37	54 - 126	26 - 256	59 - 170	40 - 166
364	pentachlorophenol	21	76 - 140		77 - 130	71 - 150
264	pentachlorophenol-13C6	49	37 - 212	18 - 412	42 - 237	29 - 254
381	phenanthrene	13	93 - 119		75 - 133	87 - 126
281	phenanthrene-d10	40	45 - 130	24 - 241	67 - 149	34 - 168
365	phenol	36	77 - 127		65 - 155	62 - 154
265	phenol-d5	161	21 - 210	ns - ns	48 - 208	ns - ns
703	alpha-picoline (Synfuel)	38	59 - 149		60 - 165	50 - 174
603	alpha-picoline-d7	138	11 - 380	ns - ns	31 - 324	ns - 608
384	pyrene	19	76 - 152		76 - 132	72 - 159
284	pyrene-d10	29	32 - 176	18 - 303	48 - 210	28 - 196
710	styrene (Appendix C)	42	53 - 221		65 - 153	48 - 244
610	styrene-d5	49	ns - 281	ns - ns	44 - 228	ns - 348
709	alpha-terpineol (Appendix C)	44	42 - 234		54 - 186	38 - 258
609	alpha-terpineol-d3	48	22 - 292	ns - 672	20 - 502	18 - 339
729	1,2,3-trichlorobenzene (4c)	69	15 - 229		60 - 167	11 - 297
629	1,2,3-trichlorobenzene-d3 (3)	57	15 - 212	ns - 592	61 - 163	10 - 282
308	1,2,4-trichlorobenzene	19	82 - 136		78 - 128	77 - 144
208	1,2,4-trichlorobenzene-d3	57	15 - 212	ns - 592	61 - 163	10 - 282
530	2,3,6-trichlorophenol (4c) (3)	30	58 - 137		56 - 180	51 - 153
731	2,4,5-trichlorophenol (4c)	30	58 - 137		56 - 180	51 - 153
631	2,4,5-trichlorophenol-d2 (3)	47	43 - 183	21 - 363	69 - 144	34 - 226
321	2,4,6-trichlorophenol	57	59 - 205		81 - 123	48 - 244
221	2,4,6-trichlorophenol-d2	47	43 - 183	21 - 363	69 - 144	34 - 226

(1) Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.

(2) ns = no specification: limit is outside the range that can be measured reliably.
(3) This compound is to be determined by internal standard; specification is derived from related compound.

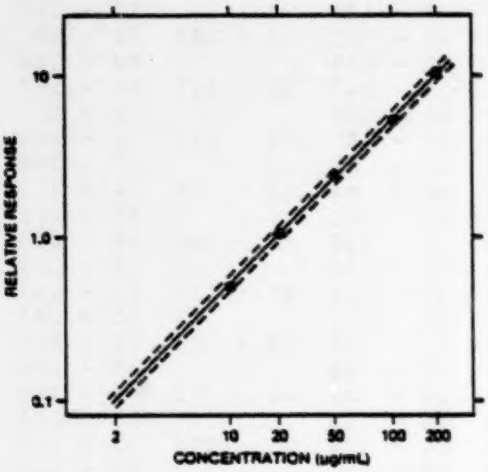

FIGURE 1 Relative Response Calibration Curve for Phenol. The Dotted Lines Enclose a ±10 Percent Error Window.

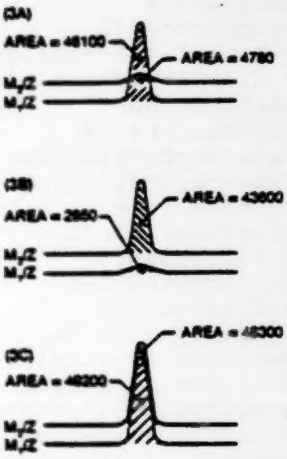

FIGURE 3 Extracted Ion Current Profiles for (3A) Unlabeled Compound, (3B) Labeled Compound, and (3C) Equal Mixture of Unlabeled and Labeled Compounds.

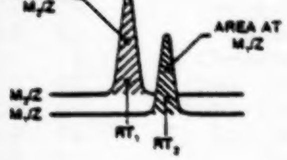

FIGURE 2 Extracted Ion Current Profiles for Chromatographically Resolved Labeled (m_2/z) and Unlabeled (m_1/z) Pairs.

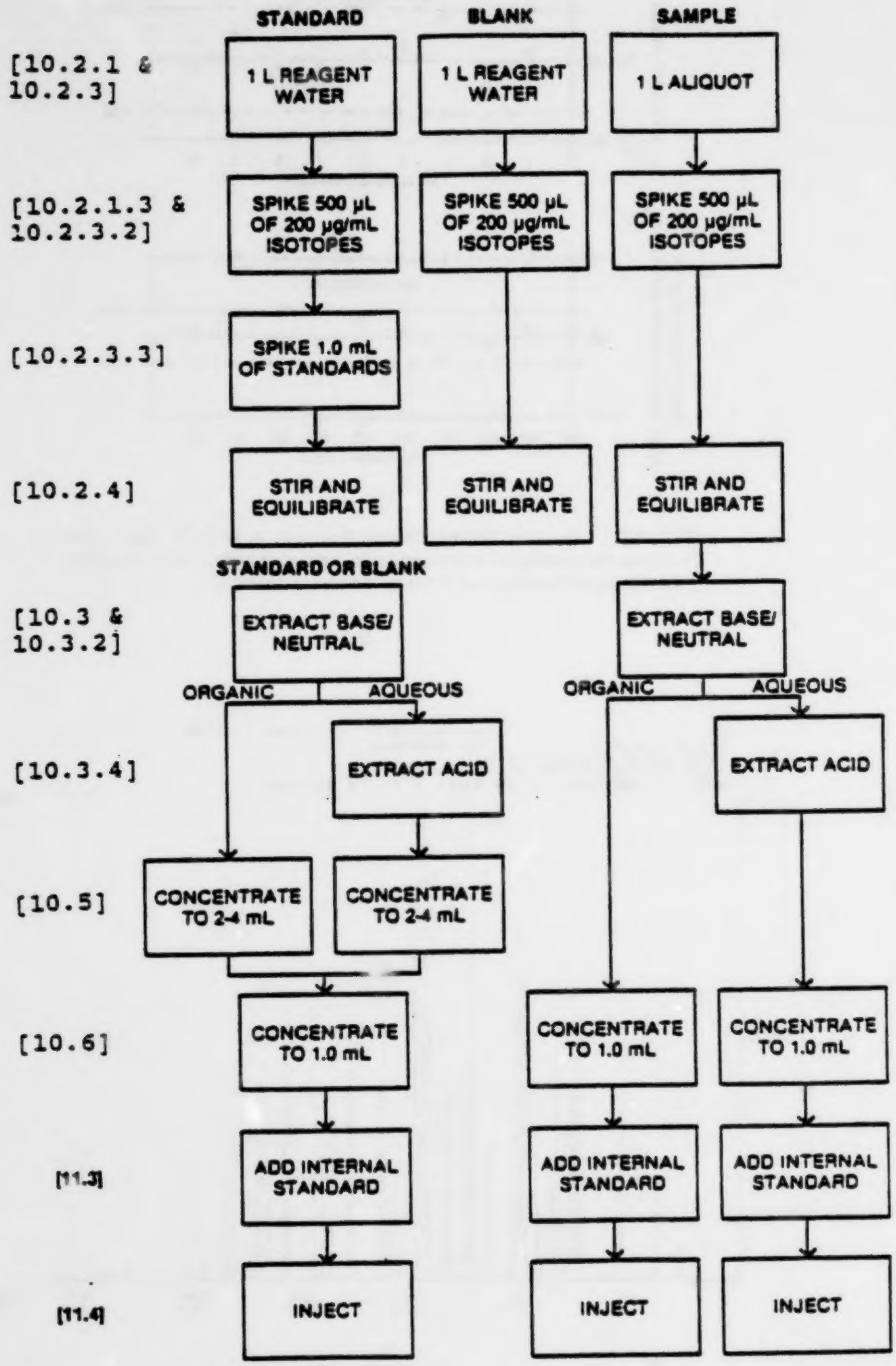

FIGURE 4 Flow Chart for Extraction/Concentration of Precision and Recovery Standard, Blank, and Sample by Method 1625. Numbers in Brackets [ ] Refer to Section Numbers in the Method.

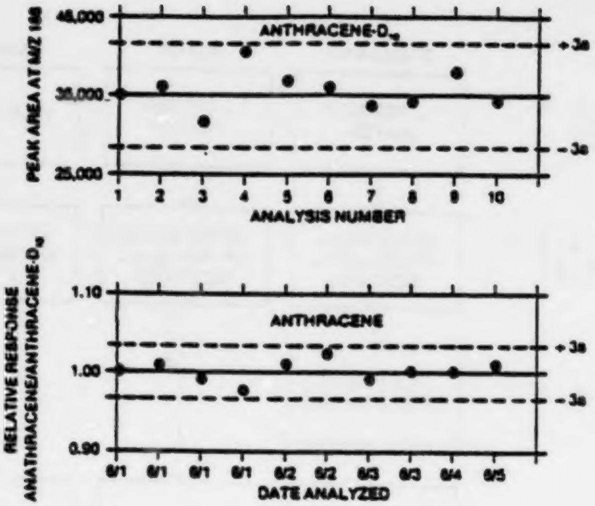

FIGURE 5 Quality Control Charts Showing Area (top graph) and Relative Response of Anthracene to Anthracene-d_{10} (lower graph) Plotted as a Function of Time or Analysis Number.

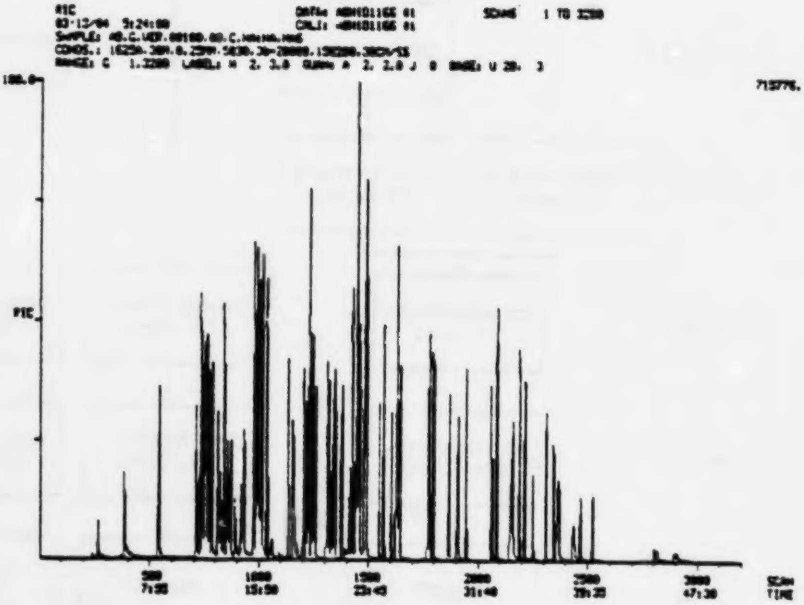

FIGURE 6 Chromatogram of Combined Acid/base/neutral Standard.

555 acetophenone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	21	43	245	49	19	50	221	51	524	52	75
61	13	62	26	63	422	65	31	73	13	74	64
75	36	76	62	77	941	78	11	89	12	91	22
105	1000	106	87	120	479	121	38				

556 4-aminobiphenyl

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	55	63	65	72	82	83	73	85	163	115	142
139	65	141	132	167	163	168	280	169	1000	170	216

557 aniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	65	41	66	42	16	46	11	47	75	50	40
51	47	52	54	53	12	54	40	61	17	62	28
63	59	64	33	65	226	66	461	74	11	78	14
91	10	92	136	93	1000	94	73				

558 o-anisidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	22	41	43	42	10	50	60	51	106	52	202
53	286	54	39	61	12	62	25	63	43	64	24
65	142	66	20	76	13	77	36	68	32	79	25
80	915	81	41	92	47	93	14	94	18	105	18
108	1000	109	55	122	123	844	124	56			

559 aramite

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	606	57	758	59	328	63	782	65	285	74	113
77	155	91	339	105	153	107	239	121	107	123	120
163	143	175	182	185	1000	187	328	191	346	197	191
319	270	334	137								

560 benzanthrone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
74	69	75	71	87	97	88	160	99	69	100	215
101	278	150	58	174	67	199	63	200	350	201	236
202	762	203	126	230	1000	231	177				

561 1,3-benzenediol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	64	41	19	52	42	43	36	49	11	50	43
51	54	52	29	53	184	54	89	55	97	61	15
62	27	63	74	64	61	65	13	68	56	69	119
71	16	81	201	82	251	95	13	109	11	110	1000
111	51										

562 benzenethiol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	128	50	149	51	205	65	175	66	505	69	114
77	161	84	259	109	316	110	1000	111	102		

563 2,3-benzofluorene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
74	52	81	69	94	143	95	253	106	60	107	205
108	491	187	75	189	90	213	233	214	60	215	987
216	1000	217	166								

943 benzoic acid

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	29	50	221	51	413	52	45	66	11	74	53
75	25	76	81	77	778	78	76	105	1000	122	868

564 benzyl alcohol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	17	59	16	50	155	51	319	52	78	53	84
61	11	62	31	63	70	64	12	65	75	74	35
75	13	76	18	77	565	78	116	79	1000	80	73
89	65	90	64	91	125	105	38	106	18	107	523
108	737	109	43								

565 2-bromochlorobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	237	50	890	51	183	73	158	74	506	75	1000
76	202	111	961	113	287	190	638	192	809	194	193

566 3-bromochlorobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	201	50	834	51	174	73	169	74	509	75	914
76	197	111	1000	113	301	190	625	192	802	194	191

567 4-chloro-2-nitroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	119	50	174	51	260	52	531	61	205	62	394
63	1000	64	315	65	192	73	290	74	105	75	156
76	127	78	152	90	724	91	253	101	232	114	312
126	766	128	234	142	211	172	915	174	289		

568 5-chloro-o-toluidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	115	51	261	52	257	53	137	77	420	78	134
79	140	89	152	106	1000	140	599	141	964	142	265
143	313										

569 4-chloroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	60	62	55	63	147	64	135	65	329	73	51
91	63	92	186	99	67	100	115	127	1000	128	81
129	292										

570 3-chloronitrobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	619	51	189	73	144	74	330	75	1000	76	169
85	101	99	258	111	851	113	266	157	424	159	137

571 o-cresol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	102	51	181	53	144	77	358	79	380	80	159
89	114	90	231	107	783	108	1000				

944 p-cresol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	136	51	224	52	106	53	196	77	420	79	308
80	145	90	122	107	822	108	1000				

572 crotoxyphos

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	633	44	448	67	42	77	70	79	41	104	100
105	484	109	21	127	1000	166	180	193	401	194	20

573 2,6-di-t-butyl-p-benzoquinone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	392	53	586	55	325	57	668	65	416	67	927
77	376	79	308	91	456	95	322	107	248	121	255
135	538	136	240	149	429	163	292	177	1000	205	203
220	410										

574 2,4-diaminotoluene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	70	42	55	51	76	52	70	53	51	61	91
67	50	77	147	78	69	93	63	94	224	104	128
105	134	106	67	121	958	122	1000	123	79		

575 1,2-dibromo-3-chloropropane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	38	59	341	51	104	61	38	75	1000	76	75
77	331	81	43	93	117	95	106	97	12	105	67
106	17	119	74	121	66	155	635	157	784	158	20
159	204	187	10								

945 3,5-dibromo-4-hydroxybenzonitrile

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
53	148	61	193	62	222	88	632	117	137	168	152
170	141	275	489	277	1000	279	451				

576 2,6-dichloro-4-nitroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	206	52	1000	61	523	62	828	63	588	73	470
65	137	89	218	90	443	97	458	124	954	126	401
133	218	160	401	176	431	178	134	206	378		

577 1,3-dichloro-2-propanol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	14	42	55	43	503	44	22	47	12	58	15
49	113	50	15	51	37	57	10	61	12	75	14
78	11	79	1000	80	25	81	310				

578 2,3-dichloroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
52	138	61	151	62	265	63	455	64	142	65	105
73	130	90	460	99	202	125	108	126	149	161	1000
163	626	165	101								

579 2,3-dichloronitrobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	220	50	257	61	150	62	120	63	173	73	336
74	976	75	743	84	351	85	166	86	125	109	1000
110	204	111	303	133	701	135	435	145	580	147	368
161	190	163	121	191	411	193	263				

946 2,6-dichlorophenol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	111	62	160	63	714	73	132	98	293	99	117
126	260	162	1000	164	613	166	101				

580 1,2:3,4-diepoxybutane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	37	41	29	42	83	43	60	55	1000	56	67
57	155	58	16	85	13						

581 3,3'-dimethoxybenzidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
65	44	79	222	85	69	93	84	107	46	115	110
122	115	158	154	186	144	201	552	229	162	244	1000
245	152										

582 dimethyl sulfone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
44	10	45	94	46	29	47	18	48	69	62	14
63	69	64	22	65	19	79	1000	81	36	94	528
96	23										

583 p-dimethylaminoazobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	483	51	181	77	447	78	120	79	147	91	109
104	142	105	190	120	1000	148	160	225	676		

584 7,12-dimethylbenzo(a)anthracene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
101	24	112	34	113	112	114	38	119	212	120	296
125	46	126	81	127	60	128	76	215	24	226	47
237	23	239	313	240	230	241	433	242	61	250	32
252	68	253	33	255	84	256	1000	257	180		

585 N,N-dimethylformamide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	58	41	79	42	497	43	115	44	1000	45	19
57	17	58	83	72	89	73	994	74	35		

586 3,6-dimethylphenanthrene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
76	113	89	129	94	179	101	142	102	151	189	388
190	193	191	430	205	246	206	1000	207	159		

587 1,3-dinitrobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	1000	51	131	63	228	64	218	74	311	75	623
76	664	92	240	122	166	168	399				

588 diphenyldisulfide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	153	51	293	65	671	59	282	77	141	109	1000
110	132	154	191	185	117	218	418				

589 ethyl methanesulfonate

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	16	43	72	45	208	48	40	59	19	63	23
64	22	65	93	79	1000	80	127	81	42	96	16
97	206	109	579	111	18	123	15	124	33		

590 ethylenethiourea

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	46	42	126	45	97	46	42	59	14	72	89
73	151	102	1000								

591 ethynylestradiol 3-methyl ether

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	155	53	101	91	157	115	143	147	226	159	132
160	115	173	199	174	313	227	1000	228	149	242	153
310	516										

592 hexachloropropene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
47	131	71	333	106	334	108	200	117	329	119	320
141	206	143	196	211	631	213	1000	215	623	217	186

947 hexanoic acid

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	627	42	535	43	214	45	186	46	19	55	128
56	90	57	102	60	1000	61	66	69	21	70	20
73	412	74	56	87	98						

593 2-isopropylnaphthalene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	100	63	111	76	157	77	129	115	147	127	131
128	216	152	133	153	184	154	114	155	1000	156	139
170	368										

594 isosafrole

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	110	51	222	63	127	77	277	78	208	103	355
104	441	131	371	132	107	135	129	161	250	162	1000

595 longifolene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
53	438	55	719	65	346	67	453	77	566	69	713
91	1000	93	611	94	546	95	404	105	614	107	475
119	394	133	338	161	568	204	172				

596 malachite green

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
118	113	126	313	165	369	208	135	209	233	210	181
237	158	253	1000	254	160	329	189	330	775	331	170

597 methapyriline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	72	45	47	53	40	58	1000	71	188	72	225
78	54	79	48	97	516	190	40	191	67		

598 methyl methanesulfonate

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	178	56	15	48	108	50	26	63	35	64	48
65	285	78	27	79	821	80	1000	81	44	82	33
95	137	109	59	110	60						

599 2-methylbenzothiozole

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	152	50	133	58	153	62	106	63	309	69	513
82	204	108	392	109	102	148	279	149	1000	150	110

900 3-methylcholanthrene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
113	58	119	55	125	83	126	305	132	99	133	122
134	160	250	56	252	322	253	271	263	59	265	106
266	50	267	192	268	1000	269	185				

901 4,4'-methylenebis(2-chloroaniline)

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
77	190	84	107	98	299	104	133	115	226	140	316
195	352	229	228	231	1000	233	227	265	171	266	631
267	144	268	358								

902 4,5-methylenephenanthrene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	50	62	55	63	95	74	69	81	145	86	53
87	60	94	255	95	659	163	80	187	213	188	137
189	900	190	1000								

903 1-methylfluorene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	66	51	87	62	57	63	137	74	64	75	85
76	196	83	135	87	53	88	78	89	203	90	58
139	54	151	73	152	124	163	57	164	58	165	1000
166	136	176	96	177	52	178	202	179	182	180	686
181	99										

904 2-methylnaphthalene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	29	51	39	57	28	58	47	62	26	63	65
65	19	69	56	70	25	71	126	74	25	75	23
76	14	77	15	86	13	87	18	89	42	113	19
114	13	115	303	116	25	126	13	139	98	140	24
141	748	142	1000	143	105						

905 1-methylphenanthrene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	54	63	86	70	62	74	51	81	52	83	164
96	132	163	55	165	217	189	165	191	532	192	1000
193	152										

906 2-(methylthio)benzothiazole

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	790	50	212	63	383	69	578	82	233	108	627
136	239	148	938	180	250	181	1000				

907 1,5-naphthalenediamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	48	65	83	77	75	79	111	103	86	118	52
130	262	131	40	141	43	157	89	158	1000	159	117

908 1,4-naphthoquinone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	445	51	62	52	52	66	69	74	189	75	205
76	590	101	51	102	613	103	52	104	550	130	433
158	1000	159	100								

909 alpha-naphthylamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	25	51	31	57	36	59	46	62	28	63	59
65	27	71	58	72	104	89	62	113	22	114	34
115	401	116	212	142	53	143	1000	144	101		

910 5-nitro-o-toluidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	194	52	159	53	121	77	766	78	176	79	619
94	168	104	120	106	691	152	1000				

911 2-nitroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	64	50	51	51	89	52	207	53	74	62	58
63	181	64	155	65	960	66	96	80	212	91	86
92	566	108	170	138	1000	139	63				

912 3-nitroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	101	52	120	53	59	62	58	63	143	64	121
65	1000	66	114	80	169	91	62	92	764	93	62
108	87	138	717	139	51						

913 4-nitroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
52	228	53	160	62	110	63	216	64	164	65	1000
66	124	80	266	92	300	108	636	138	520		

914 4-nitrobiphenyl

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	131	63	104	76	179	115	134	141	277	151	259
152	902	153	284	169	374	199	1000	200	125		

915 N-nitroso-di-n-butylamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	1000	42	536	43	570	44	313	55	129	56	167
57	994	84	985	86	103	99	197	115	158	116	237
158	161										

916 N-nitrosodiethylamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	170	42	079	43	69	44	1000	45	20	54	18
56	525	57	492	70	24	71	28	85	25	87	31
102	807	103	35								

917 N-nitrosomethylethylamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	117	42	1000	43	667	44	26	54	17	56	189
57	99	59	13	71	60	73	57	88	772	89	20

918 N-nitrosomethylphenylamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	181	51	434	52	104	63	110	77	1000	78	194
79	331	104	147	106	673	107	220	212	137		

919 N-nitrosomorpholine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	181	42	192	43	52	44	17	54	85	55	95
56	1000	57	49	85	13	86	333	87	14	116	337

920 N-nitrosopiperidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	320	42	1000	43	43	51	14	52	12	53	32
54	58	55	444	56	224	57	17	67	21	82	26
83	28	84	47	114	491	115	26				

921 pentachlorobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
73	160	108	239	125	102	178	102	213	179	215	218
217	106	248	648	250	1000	252	642	254	199		

922 pentachloroethane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
47	203	60	398	62	119	83	378	85	218	94	114
95	165	117	1000	119	979	121	306	130	293	132	272
165	716	167	901	169	422						

923 pentamethylbenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	126	53	84	63	61	65	99	77	145	79	64
91	218	105	128	115	120	117	91	133	1000	134	105
147	60	148	420								

924 perylene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
74	33	111	43	112	70	113	111	124	132	125	251
126	243	224	49	248	75	249	52	250	284	251	86
252	1000	253	219								

925 phenacetin

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
43	443	51	33	52	112	53	164	63	39	64	30
65	47	79	31	80	179	31	154	108	1000	109	196
110	50	137	461	138	40	179	672	180	64		

926 phenothiazine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	145	51	120	63	134	69	190	100	128	154	149
166	240	167	607	198	186	199	1000	200	143		

927 1-phenylnaphthalene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	132	51	156	63	148	74	124	75	142	76	136
87	101	88	183	89	162	100	155	101	527	102	111
200	144	201	136	202	643	203	1000	204	999	205	159

928 2-phenylnaphthalene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	108	63	101	76	136	88	133	89	158	101	333
102	188	202	398	203	270	204	1000	205	157		

929 pronamide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	270	66	109	74	112	75	137	84	194	109	186
145	334	147	198	173	1000	175	615	254	133	255	211
256	102	257	122								

930 pyridine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	45	48	11	49	62	50	324	51	414	52	879
53	112	54	12	55	16	75	21	76	19	77	22
78	151	79	1000	80	101	81	58				

931 safrole

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	132	51	369	63	108	77	391	78	228	103	348
104	477	105	130	131	437	132	166	161	298	162	1000
163	109										

932 squalene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
53	62	55	94	67	105	68	119	69	1000	70	57
79	43	81	465	82	52	93	70	95	104	107	43
109	47	121	46	137	41						

933 1,2,4,5-tetrachlorobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
47	125	49	176	61	127	72	183	73	332	74	448
84	197	108	284	109	231	143	194	145	117	179	237
181	224	214	791	216	1000	218	482	220	101		

948 2,3,4,6-tetrachlorophenol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
61	234	65	167	66	105	83	134	84	178	96	202
97	107	131	463	133	270	166	298	168	273	194	168
196	164	230	793	232	1000	234	471				

934 thianaphthene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	80	50	91	51	65	62	82	63	162	67	78
69	139	74	55	89	191	90	136	108	82	134	1000
135	104	136	52								

935 thioacetamide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	225	42	485	43	44	46	18	57	36	58	93
59	165	60	437	75	1000	76	25	77	43		

936 thioxanthone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	262	63	180	69	320	74	116	69	176	82	121
92	188	108	129	139	385	152	227	183	112	184	951
185	137	212	1000	213	145						

937 o-toluidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	51	41	38	42	35	49	10	50	88	51	169
52	164	53	192	53	86	62	26	63	68	64	30
65	59	66	24	74	19	65	14	76	21	77	313
78	113	79	243	80	80	89	107	90	76	91	52
104	45	106	1000	107	90						

938 1,2,3-trimethoxybenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	257	51	459	52	139	53	276	63	112	65	341
67	114	77	246	79	132	82	117	93	483	95	801
107	190	108	144	110	898	125	578	153	759	168	1000

939 2,4,5-trimethylaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	80	52	58	51	63	53	66	65	150	67	74
79	62	91	167	93	51	117	54	118	65	119	93
120	1000	121	87	134	670	135	978	136	99		

940 triphenylene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
74	52	87	55	100	107	101	108	112	131	113	244
114	181	200	67	202	56	224	84	225	56	226	313
227	132	228	1000	229	184						

941 tripropylene glycol methyl ether

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	492	46	15	47	19	55	17	57	68	58	43
59	1000	60	34	71	16	72	44	73	363	74	232
103	57	117	92	161	21						

942 1,3,5-trithiane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
46	1000	47	150	48	98	59	93	60	76	64	136
73	102	91	92	92	111	110	58	138	259		

ATTACHMENT 8

INSTRUCTIONS FOR USE OF LABELED COMPOUND SOLUTIONS

Instructions for Use of Labeled Compound Solutions

1 Scope and application. These instructions define use of stable isotope labeled pollutants provided by EPA for the analysis of water and wastewater.

1.1 The solutions of mixtures listed in tables 1 through 11 for volatiles, semi-volatiles (acids and base/neutrals), and Appendix C compounds permit analysis of nearly all non-pesticide organic priority pollutants by isotope dilution methods.

1.2 These instructions were written explicitly for use with Revision C of EPA Methods 1624 and 1625. The numbers in brackets [ ] refer to specific section numbers in these methods.

1.3 For accurate isotope dilution analyses, it is imperative that identical concentrations of the labeled compounds be used for calibration and for spiking into water samples.

1.4 Analysts unfamiliar with isotope dilution and/or GCMS methods for analysis of organic priority pollutants in waters should review references 1-3 before proceeding.

1.5 EPA may revise these instructions to account for new labeled compounds, new compound concentrations, or new mixtures of compounds.

2 Description of solutions

2.1 The solutions listed in tables 1-11 are contained in snap-top, flame sealed ampuls. One mL is contained in each ampule; however, this volume should not be relied upon for precise measurement of solution concentrations or total amounts of compounds present (see section 1.3).

2.1.1 EPA has discontinued the use of Mininert valves with these solutions. It is the laboratory's responsibility to provide valved ampules so that changes in concentrations of the solutions by evaporative loss are minimized.

2.2 A data sheet is attached for each solution (Tables 1-11) giving exact concentrations of the constituents. These concentrations are accurate to ± 5 percent at the time the solution was prepared. Solution concentrations are to be monitored for changes by the laboratory [1624C: 6.5.4; 1625C: 6.15].

2.2.1 EPA may change the concentration and/or the mixture and/or number of compounds in these solutions; therefore, the laboratory shall observe the compounds and concentrations in each and every solution prior to use, to insure consistency with the compounds and concentrations in the methods.

2.3 Volatiles

2.3.1 Four solutions are provided: "purgeables/volatiles E.1, F, G and H." (Tables 1-4)

2.3.1.1 The nominal concentration of solution G is 250 ug/mL in methanol-d_4.

2.3.1.2 Nominal concentrations of solutions E.1, F, and H are 50 ug/mL in methanol-d_4.

2.4 Semi-volatiles--acids and base/neutrals.

2.4.1 Nominal concentrations are 5 mg/mL in benzene-d_6.

2.4.2 Acids--one solution is provided: "acid extractables 3. (Table 5)"

2.4.3 Base/neutrals--six solutions are provided: "base/neutrals 4.1, 4.3, 6.2, 6.3" (Tables 6-9) and "base/ neutrals 5.1 (Table 10) " which contains all of the priority pollutant, Appendix C, paragraph 4C, and synfuels amine compounds.

2.4.4 Appendix C--one solution is provided: "semi-volatiles-1/Appendix C" (Table 11) , which contains the nonamine Appendix C and synfuel base/neutral compounds.

NOTE: In preparing this set of labeled compounds, it was discovered that the alpha-terpineol-d3 was mislabeled, and was actually dibenzothiophene-d8. Therefore, alpha-terpineol-d3 is not contained in this solution, and dibenzothiophene-d8 is contained at a concentration of 10 mg/mL.

2.5 The ampuls shall be opened per the procedure in section 5.

3 Storage

3.1 Unopened ampuls--store in a freezer at -20° to -10°C until ready for use. Do not open until required.

3.2 Opened ampuls--store in a refrigerator at 0° to 4°C. Mininert valved ampules should not be stored at temperatures below 0°C as the Teflon seal on the ampule top is not reliable below this temperature.

3.3 If the solutions are transferred from the ampul(s) to a vial or other container, store below 4°C at as low a temperature as practicable, consistent with a reliable seal.

3.4 Except for unopened ampuls, the storage appliance (e.g., refrigerator, freezer) for volatile solutions must be free of solvent vapors which would interfere with the analysis of either the labeled compounds or priority pollutants. Do not store solutions of standards in methylene chloride, benzene, dioxane or other solvents (except dilute solutions in methanol) in the same appliance as the volatiles solutions.

4 Obtaining mass spectra [1624C: 7.2; 1625C: 7.2]

4.1 If the laboratory does not have a mass spectrum of the specific labeled compounds contained in the solutions (tables 1-11), a spectrum should be obtained prior to mixing the solutions (section 6) to minimize the necessity to deconvolute multiple spectra.

4.2 Open the ampul per the procedure in section 5.

4.3 Inject 0.1 to 2 uL (or an amount as required) of the solution into the GC. Alternatively, purge the volatiles solution from 5 mL reagent water. Analyze the solution under the GC conditions for the method [1624C: table 3; 1625C: tables 5 and 6] so that retention time data are obtained.

5 Opening an ampul--open in a hood, while wearing gloves.

5.1 Materials required.

5.1.1 Needle-nosed pliers or tweezers.

5.1.2 Mininert valves--to be used if the solution is to be retained in a valved ampule.

5.1.3 Syringe--1 mL, cleaned, with 2 inch needle, to be used if solution is to be removed from ampule.

5.2 Bring the ampul to room temperature. Inspect for damage and possible loss of solvent. Notify the Sample Control Center if loss or change has occurred.

5.3 If the solution is to be withdrawn from the ampule, be certain that all precipitate has redissolved. Warm to no more than 35°C (if required) before opening.

5.4 If liquid has collected in the flame seal tube area of the ampul, tap the tube gently with a finger to dislodge the solution from this area.

5.5 If the Mininert valve assembly is to be used, inspect the valve for burrs or other defects which might preclude its immediate use. If the solution is to be withdrawn from the ampul, have the necessary syringe(s) and vial(s) available so that loss by evaporation will be minimized.

5.6 Snap the top on the ampul. Inspect the broken edge remaining on the ampul body. If any portion of the edge protrudes above the rim of the ampul, this edge will cut the bottom of the Mininert valve. Carefully break off and remove this protruding edge using needle-nose pliers or tweezers. Thread the Mininert valve onto the ampul and tighten to form a seal.

5.7 Mark the solution level on the ampul label. Scrape off a portion of the label if required for viewing the meniscus..

5.8 Document the date and operations in a standards log book.

6 Mixing and diluting. If a spectrum of each labeled compound has not been obtained, refer to section 4.

6.1 The most efficient and precise use of the labeled solutions results from mixing and diluting (if required) to form single solutions for sample spiking [1624C: 6.6; 1625C: 6.8], calibration [1624C: 6.7.1; 1625C: 6.13], and for performance verification [1624C: 6.7.2 and 11.1; 1625C: 6.14 and 12.1].

6.2 Materials required.

6.2.1 Syringes--1 mL, cleaned, with 2 inch needles.

6.2.2 Vials--5 and 40 mL, cleaned, with Teflon-lined screw caps.

6.2.3 Volumetric flask--25 mL, cleaned, ground glass stoppered.

6.2.4 Solvents--methylene chloride-d_2, methanol-d_4, benzene-d_6, and/or p-dioxane-d_8. If p-dioxane-d_8 is used, peroxides must be less than 25 ppm.

6.3 The following instructions apply if all compounds are used at approximately the same concentration in standards, blanks, and samples. If varied concentrations are to be used, the volumes of solutions which are mixed need to be suitably adjusted.

6.4 Volatiles

NOTE: These instructions are based on the assumption that the volatile gases and water soluble compounds (1624C: 6.6) will be employed at five times the level (i.e., at 50, 100, 200, 500, and 1000 ug/L) of the other volatile labeled compounds. If the gases and water soluble compounds are to be employed at the same level (i.e., at 10, 20, 50, 100, and 200 ug/L) as the other volatile labeled compounds, the "purgeables/volatiles G" solution must be diluted by a factor of five (5) prior to mixing with the remaining purgeables/volatiles E.1, F, and H solutions.

6.4.1 Open one ampul at a time per the instructions in section 5. Withdraw the total volume of solution in a one mL syringe. If less than 0.8 mL is present do not use the solution, repackage it in the ampul and seal with the Mininert valve. Report the shortage to the Sample Control Center.

6.4.2 Place the solution in a 5 mL vial and cap immediately. Add the other three solutions (table 1) to the vial and cap tightly.

6.4.3 The final nominal solution concentration in the vial will be 62.5 ug/mL (250 ug/mL divided by 4) for the gases and water soluble compounds and 12.5 ug/mL (50 ug/mL divided by 4) for the remaining volatile compounds.

NOTE: If the "purgeables/volatiles G" solution was divided by a factor of 5 per the "NOTE" under section 6.4 above, all compounds in the vial will be at a concentration of 12.5 ug/mL.

6.5 Semi-volatiles

6.5.1 Open one ampul at a time per the instructions in section 5. Observe the caution in 5.3. Withdraw the total volume of solution in a one mL syringe. If less than 0.8 mL is present do not use the solution, repackage it in the ampul and seal with the Minimert valve. Report the shortage to the Sample Control Center.

6.5.2 Place the solution in a 25 mL ground glass stoppered volumetric flask. Add the other solutions (tables 2-4) to the flask. Bring to the mark with benzene-d_6, p-dioxane-d_8, or a mixture of these two solvents. Note: it was not known at the time of writing of these instructions if all compounds would be soluble in methylene chloride-d_2 at the dilute (200 ug/L) level. The laboratory should test this solubility on unlabeled compounds if use of methylene chloride is desired.

6.5.3 Transfer the solution to a 40 mL vial with Teflon-lined screw cap.

6.5.4 The final solution concentration will be nominal 200 ug/mL [1625C: 6.8] (5000 ug/mL divided by 25). Spike 0.5 mL of this solution into each sample aliquot (1625C: 10.2.1.3, 10.2.2.6, or 10.2.5.6).

6.6 Label the above solution and place a mark at the solvent level.

6.7 Document the date and operations in a standards log book.

7 Questions comments/problems.

7.1 Contact the Sample Control Center if difficulties arise in the use of the labeled compound solutions or these instructions.

8 References

8.1 Methods 1624 and 1625, Revisions C, USEPA, Industrial Technology Division, WH-552, 401 M Street, SW, Washington, D.C. 20460.

8.2 Methods 601-625, July 1982 Revisions, USEPA, EMSL, Cincinnati, OH 45268, 49 FR 43234 (26 October, 1984).

8.3 "Performance Tests for the Evaluation of Computerized Gas Chromatography/Mass Spectrometry Equipment and Laboratories," USEPA, EMSL Cincinnati, OH 45268, EPA-600/4-80-025 (April 1980).

Table 1

Lot No.0-641

Packaging sub-lot 1970-1

MW-2945 PURGEABLES/VOLATILES-E .1

(50 µg each component/1 ml. methanol-d_4 solution)

EPA No.	Compound	MSD No.	Chemical Purity %	Isotopic Purity Atom %[1]	Concentration in solution µg/ml[2]
6V	Carbon-^{13}C Tetrachloride	MS-1312	≥ 99	94.31	50
7V	Chlorobenzene-d_5	MD-786	≥ 99	99.61	50
23V	Chloroform-^{13}C	MS-1318	≥ 99	93.9	50
13V	1,1-Dichloroethane-2,2,2-d_3	MD-1152	≥ 98	98.77	50
29V	1,1-Dichloroethylene-d_2	MD-2201	≥ 99	98.43	50
44V	Dichloromethane-d_2	MD-53	≥ 99	99.86	50
32V	1,2-Dichloropropane-d_6	MD-2363	≥ 99	99.23	50
14V	1,1,2-Trichloroethane-1,2-$^{13}C_2$	MS-2346	≥ 99	96.6	50

Footnotes: 1. Isotopic purity, atom % D or atom % ^{13}C, determined by NMR and/or MS ± 0.2%.

2. Actual concentration, confidence limit ± 5%.

Table 2

Lot No. 0-642

Packaging sub-lot 1971-1

MW- 2946 PURGEABLES/VOLATILES-F

(50 µg each component/1 mL methanol-d_4 solution)

EPA No.	Compound	MSD No.	Chemical Purity %	Isotopic Purity Atom %	Concentration in solution µg/ml
4V	Benzene-d_6	MD-6	>99.5	99.7	50
47V	Bromoform-^{13}C	MS-2313	>99	92.5	50
10V	1,2-Dichloroethane-d_4	MD-103	>99	99.22	50
38V	Ethylbenzene-d_{10}	MD-1766	>98	98.34	50
15V	1,1,2,2-Tetrachloroethane-d_2	MD-1416	>99	99.56	50
86V	Toluene-d_8	MD-40	>99	99.64	50
11V	1,1,1-Trichloroethane-d_3	MD-1150	>99	97.15	50

Footnotes: 1. Isotopic purity, atom % D or atom % ^{13}C, determined by NMR and/or MS, ± 0.2%

2. Actual concentration, confidence limit ± 5%

Table 3

MW-2947 PURGEABLES/VOLATILES-C

Lot No. 0-643

(250 μg each component/1 mL methanol-d_4 solution)

Packaging sub-lot 1972-1

EPA No.	Compound	MSD No.	Chemical Purity %	Isotopic Purity Atom %	Concentration in solution μg/ml
516V	Acetone-d_6	MD-2	>99.5	99.6	250
2V	Acrolein-d_4 *	MD-2604	≥99	97.7	250
3V	Acrylonitrile-d_3	MD-1455	≥99	99.01	250
46V	Bromomethane-d_3	MD-23	≥98	99.5	250
514V	2-Butanone-4,4,4-d_3	MD-2402	≥99	99.27	250
16V	Chloroethane-d_5	MD-334	≥99	>99	250
45V	Chloromethane-d_3	MD-324	≥99	99.55	250
515V	Diethyl-d_{10} Ether	MD-267	>99	99.1	250
88V	Vinyl-d_3 Chloride *	MD-965	≥99	99.75	250

Footnotes:
1. Isotopic purity, atom % D, determined by NMR and/or MS, ± 0.2%
2. Actual concentration, confidence limit ± 5%
3. * Both products stabilized with hydroquinone

Table 4

MW- 2948 PURGEABLES/VOLATILES-II

(50 µg each component/1 mL methanol-d_4 solution)

Lot No. 0-644

Packaging sub-lot 1964-I

EPA No.	Compound	MSD No.	Chemical Purity %	Isotopic Purity Atom %	Concentration in solution µg/ml
48V	Bromodichloromethane-^{13}C	MS-2368	≥99	≥92.57	50
51V	Chlorodibromomethane-^{13}C	MS-2364	≥99	92.33	50
30V	1,2-Dichloroethylene-1,2-d_2 (cis/trans mixture)	MD-2526	≥99	99.57	50
33V	1,3-Dichloropropene-d_4 (cis/trans mixture)	MD-2669	≥99	99.27	50
527V	p-Dioxane-d_8	MD-426	≥99	99.03	50
85V	Tetrachloroethylene-1,2-$^{13}C_2$	MS-2411	≥99	98.94	50
87V	1,1,2-Trichloroethylene-1,2-$^{13}C_2$	MS-2410	≥99	99.28	50

Footnotes: Isotopic purity, atom % D or atom % ^{13}C, determined by NMR and/or MS ± 0.2%

Actual concentration, confidence limit ± 5%

Table 5

MW-2949 ACID EXTRACTABLES - 3

(5 mg each component/1 mL benzene-d_6 solution)

Lot No. 0-645

Packaging sub-lot 2155-I

EPA No.	Compound	MSD No.	Chemical Purity %	Isotopic Purity Atom %[1]	Concentration in solution mg/ml
22A	4-Chloro-3-methylphenol-2,6-d_2	MD-2355	> 99.5	98.2	5.0
24A	2-Chlorophenol-3,4,5,6-d_4	MD-2280	99	98.5	5.0
31A	2,4-Dichlorophenol-3,5,6-d_3	MD-2281	99.5	99.0	5.0
34A	2,4-Dimethylphenol-3,5,6-d_3	MD-2284	98	98.2	5.0
60A	4,6-Dinitro-2-methylphenol-3,5-d_2	MD-2357	> 99.5	98.7	5.0
59A	2,4-Dinitrophenol-3,5,6-d_3	MD-2285	≥ 99	97.9	5.0
57A	2-Nitrophenol-3,4,5,6-d_4	MD-2290	99.5	99.0	5.0
58A	4-Nitrophenol-2,3,4,5-d_4	MD-2356	99	98.7	5.0
64A	Pentachlorophenol-$^{13}C_6$	MS-2293	> 99	98.4	5.0
65A	Phenol-2,3,4,5,6-d_5	MD-1502	≥ 98.5	98.8	5.0
21A	2,4,6-Trichlorophenol-3,5-d_2	MD-2279	> 99.5	98.2	5.0
531A	2,4,5-Trichlorophenol-3,6-d_2	MD-2935	99	96.7	5.0

Footnotes: 1. Isotopic purity, atom % D or atom % ^{13}C, determined by NMR and/or MS, ± 0.2%

2. Actual concentration, confidence limit ± 5%.

Table 6

MW- 2764 BASE NEUTRALS-4.1

(5 mg each component/1 mL benzene-d_6 solution)

Lot No. 0-635

Packaging sub-lot 1965-I

EPA No.	Compound	MSD No.	Chemical Purity %	Isotopic Purity Atom %	Concentration in solution mg/ml
77B	Acenaphthylene-d_8	MD-128	≥ 99	99.4	5.0
74B	Benzo(b)fluoranthene-d_{12}	MD-2360	≥ 98	99.39	5.0
79B	Benzo(ghi)perylene-d_{12}	MD-830	98	98.5	5.0
73B	Benzo(a)pyrene-d_{12}	MD-1956	≥ 97	99.2	5.0
42B	Bis(2-chloroisopropyl)-d_{12} Ether	MD-2702	≥ 98	99.2	5.0
26B	1,3-Dichlorobenzene-d_4	MD-2405	≥ 99	98.34	5.0
35B	2,4-Dinitrotoluene-3,5,6-d_3	MD-2407	> 99	98.17	5.0
39B	Fluoranthene-d_{10}	MD-2361	> 98	99.34	5.0
52B	Hexachloro-1,3-butadiene-$^{13}C_4$	MS-2408	99	98.74	5.0
53B	Hexachlorocyclopentadiene-1,2,3,4-$^{13}C_4$	MS-2710	≥ 95	96.9	5.0
81B	Phenanthrene-d_{10}	MD-120	> 99.5	98.52	5.0

Footnotes: 1. Isotopic purity, atom % D or atom % ^{13}C, determined by NMR and/or MS, ± 0.2%.

2. Actual concentration, confidence limit ± 5%.

Table 7

MW- 2942 BASE NEUTRALS-4.3

Lot No. 0-636

(5 mg each component/1 ml. benzene-d_6 solution)

Packaging sub-lot 1966-I

EPA No.	Compound	MSD No.	Chemical Purity %	Isotopic Purity Atom %	Concentration in solution mg/ml
41B	4-Bromophenyl Phenyl-d_5 Ether	MD-2936	> 98	96.95	5.0
20B	2-Chloronaphthalene-d_7	MD-2462	> 99	98.54	5.0
40B	4-Chlorophenyl Phenyl-d_5 Ether	MD-2312	> 99	97.0	5.0
68B	Di-n-butyl Phthalate-3,4,5,6-d_4	MD-2310	> 99	98.64	5.0
70B	Diethyl Phthalate-3,4,5,6-d_4	MD-2726	> 99	99.54	5.0
69B	Di-n-octyl Phthalate-3,4,5,6-d_4	MD-2291	> 99	98.62	5.0
9B	Hexachlorobenzene-$^{13}C_6$	MS-2354	> 99	98.3	5.0
12B	Hexachloroethane-1-^{13}C	MS-2406	> 99	92.52	5.0
54B	Isophorone-d_8	MD-2304	> 99.5	99.4	5.0
8B	1,2,4-Trichlorobenzene-3,5,6-d_3	MD-2706	> 99	98.73	5.0

Footnotes:

1. Isotopic purity, atom % D or atom % ^{13}C, determined by NMR and/or MS, ± 0.2%.
2. Actual concentration, confidence limit ± 5%.

Table 8

MW- 2768 BASE NEUTRALS-6.2

Lot No. 0-639

(5 mg each component/1 mL benzene-d_6 solution)

Packaging sub-lot 2154-1

EPA No.	Compound	MSD No.	Chemical Purity %	Isotopic Purity Atom %	Concentration in solution mg/ml
1B	Acenaphthene-d_{10}	MD-42	> 99.5	97.97	5.0
78B	Anthracene-d_{10}	MD-46	> 99	99.13	5.0
75B	Benzo(k)fluoranthene-d_{12}	MD-2362	> 97.5	99.53	5.0
18B	Bis(2-chloroethyl)-d_8 Ether	MD-2479	> 98	98.17	5.0
76B	Chrysene-d_{12}	MD-402	> 99.5	99.05	5.0
80B	Fluorene-d_{10}	MD-1298	> 98	98.98	5.0
55B	Naphthalene-d_8	MD-26	99.5	99.52	5.0
84B	Pyrene-d_{10}	MD-363	> 99.5	98.9	5.0

Footnotes:
1. Isotopic purity, atom % D, determined by NMR and/or MS, ± 0.2%.
2. Actual concentration, confidence limit ± 5%.

Table 9

MW- 2944 BASE NEUTRALS-6.3

(5 mg each component/1 mL benzene-d_6 solution)

Lot No. 0-638

Packaging sub-lot 1968-I

EPA No.	Compound	MSD No.	Chemical Purity %	Isotopic Purity Atom %	Concentration in solution mg/ml
72B	Benz(a)anthracene-d_{12}	MD-364	> 99	98.7	5.0
43B	Bis(2-chloroethoxy)-d_8-methane	MD-2939	> 98	99.05	5.0
66B	Bis(2-ethylhexyl)Phthalate-3,4,5,6-d_4	MD-2306	98.7	98.84	5.0
67B	Butyl Benzyl Phthalate-3,4,5,6-d_4	MD-2940	98	98.5	5.0
82B	Dibenzo(a,h)anthracene-d_{14}	MD-520	> 98	99.08	5.0
25B	1,2-Dichlorobenzene-d_4	MD-1191	> 99	98.31	5.0
27B	1,4-Dichlorobenzene-d_4	MD-1034	> 99	98.55	5.0
71B	Dimethyl Phthalate-3,4,5,6-d_4	MD-2305	> 99	98.62	5.0
36B	2,6-Dinitrotoluene-α,α,α-d_3	MD-2359	> 99.5	99.32	5.0
56B	Nitrobenzene-d_5	MD-27	99	99.44	5.0
529B	1,2,3-Trichlorobenzene-4,5,6-d_3	MD-2511	> 98	98.18	5.0

Footnotes: 1. Isotopic purity, atom % D, determined by NMR and/or MS, ± 0.2%

2. Actual concentration, confidence limit ± 5%

Table 10

MW-2943 BASE NEUTRALS-5.1

(5 mg each component/1 mL benzene-d_6 solution)

Lot No. 0-637

Packaging sub-lot 1967-I

EPA No.	Compound	MSD No.	Chemical Purity %	Isotopic Purity Atom %	Concentration in solution mg/ml
5B	Benzidine-d_8 (rings-d_8)	MD-2330	> 98	99.3	5.0
528B	Carbazole-d_8,NH	MD-2700	> 98	99	5.0
28B	3,3'-Dichlorobenzidine-d_6 (rings-d_6)	MD-2703	98	99.3	5.0
507B	Diphenyl-d_{10}-amine	MD-2704	≥ 99	98.4	5.0
37B	1,2-Diphenyl-d_{10}-hydrazine	MD-2705	> 99	98.0	5.0
503B	2-Methylpyridine-d_7 (α-Picoline)	MD-2320	≥ 99	99.0	5.0
502B	2-Naphthyl-d_7-amine	MD-191	> 98	98.0	5.0
61B	N-Nitrosodimethyl-d_6-amine	MD-2937	99	99.6	5.0
62B	N-Nitrosodiphenyl-2,2',4,4',6,6'-d_6-amine	MD-2311	98.1	98.6	5.0
63B	N-Nitrosodi-n-propyl-d_{14}-amine	MD-2938	99	98.8	5.0

Footnotes: 1. Isotopic purity, atom % D, determined by NMR and/or MS ± 0.2%

2. Actual concentration, confidence limit ± 5%

Table 11

MW- 2769 SEMI-VOLATILES-I/APPENDIX C

Lot No. 0-599

Packaging sub-lot 1969-I

(5 mg each component/1 mL benzene-d_6 solution)

EPA No.	Compound	MSD No.	Chemical Purity %	Isotopic Purity Atom %	Concentration in solution mg/ml
513B	p-Cymene-d_{14}	MD-2709	≥ 99	99.4	5.0
517B	n-Decane-d_{22}	MD-960	≥ 99	99.1	5.0
505B	Dibenzofuran-d_8	MD-2316	> 98.5	99.1	5.0
504B	Dibenzothiophene-d_8	MD-2315	> 99	99.5	10.0*
512B	Diphenyl-d_{10}	MD-208	> 99.6	99.4	5.0
508B	Diphenyl-d_{10} Ether	MD-373	≥ 99	99.3	5.0
506B	n-Dodecane-d_{26}	MD-882	≥ 99	98.2	5.0
521B	n-Eicosane-d_{42}	MD-1532	≥ 99	98.8	5.0
519B	n-Hexadecane-d_{34}	MD-821	≥ 99	98.7	5.0
510B	Styrene-2,3,4,5,6-d_5	MD-126	≥ 99	> 98.4	5.0
509B	α-Terpineol-d_3	MD-2707	supplied	by EPA, Washington DC.*	0.0
523B	n-Tetracosane-d_{10}	MD-883	≥ 99	99.5	5.0
526B	n-Triacontane-d_{62}	MD-2708	≥ 99	99.2	5.0

Footnotes: Isotopic purity, atom % D, determined by NMR and/or MS ± 0.2%

Actual concentration, confidence limit ± 5%

* Material supplied to MSD by EPA labelled as "α-Terpineol-d_3" turned out to be dibenzothiophene-d_8. (telecon with Dale Rushneck, December 20, 1984)

ATTACHMENT 9

QUANTITATION REPORTS ON MAGNETIC TAPE

NOTE: Quantitation report types have been simplified. Please see section 3.1.3 on the sample line for further information.

Quantitation Reports on Magnetic Tape

1. Tape Characteristics

a. Tape - 9 Track; 800/1600 BPI; 600, 1200, or 2400 foot reels

b. Code - ASCII

c. Labels - no internal labels

d. Blocksize - 800 decimal words/block or bytes/block

2. File Characteristics

a. Each quantitation report represents a file on the submitted tape. A tape will contain multiple files. Each of these files must end with an end of file (tape mark).

b. Each line of the quantitation report constitutes a record. Records/lines can be variable in length from one to 80 characters. Each record/line must end with a carriage return (Octal 15 or Hexidecimal OD). A blank line is interpreted as two consecutive carriage returns. A form feed character (Octal 14 or Hexidecimal OC) must be used after the last record in the file to signify the end of all records.

c. Records/lines must be combined into fixed length blocks of 800 bytes in length. Blocks should not include any prefixes or postfixes. Records may span blocks. A file will consist of multiple blocks. Blocks are separated on the tape by inter-record gaps.

3. Data Format

The quantitation report is divided into four basic sections for information reporting. The sections in the order in which they appear within the quantitation report are called:

- Header Section
- Compound List Section
- Technical Data Section
- Reference Data Section

Each of these sections must be present in each quantitation report submitted on tape. The absence of any one of these sections from a quantitation report is cause for the non acceptance of the quantitation report. Tables 1A, 1B and 1C provide examples of quantitation reports for volatiles, semi-volatiles and dioxins, respectively. These tables are examples only, and are not meant to show the exact format in which data are to be submitted.

3.1 Header Section

The header section provides descriptive information about the analysis and the conditions under which it was performed. This section consists of three mandatory lines of descriptive information and one optional line. The four lines and the reporting requirements for each are identified as follows.

Line Identification	Reporting Requirement
Title Line	Mandatory
Date/Time Analyzed Line	Mandatory
Sample Line	Mandatory
Condition Line	Optional

Although the mandatory lines of information must be present in each header section, there is no actual limit as to the total number of lines that may be present. Laboratories are permitted to use this section to record any additional information deemed necessary to properly identify the analysis. The mandatory and optional lines however must appear in the order specified above, but additional lines may be interspersed between them. The complete specifications for each of these four lines is provided in subsequent paragraphs. Examples of the Sample and Condition Lines in the Header Section for methods 1624C, 1625C and 613 are given at the end of each respective section.

3.1.1 Title Lines

This must be the first line within the header section and as such represents the first line of each quantitation report submitted. Only one title line is permitted within the Header Section. The line must contain the value QUANTITATION REPORT starting in position one (left most position) of the line. Other information may appear after this value on the line but must be separated from the value by at least one space (blank).

3.1.2 Date/Time Analyzed Line

This line must contain the date and time that the analysis was performed. This line must preceed any other date lines within the header section. The format of this line is as follows:

Line Position	Data Element	Format
1-8	Date Analyzed	MM/DD/YY MM = month; 01-12 DD = day; 01-31 YY = year; 83-99
9	Field Delimiter	Space
10-17	Time Analyzed	HH:MM:SS HH = hour; 00-24 MM = minute; 00-59 SS = second; 01-59

3.1.3 Sample Line

The Sample line contains the following data elements. Data elements are recorded on the line in the order specified. Data elements are separate from each other by means of a comma (,). The end of the data element list is signified by a dollar sign ($). The specifications for the sample line are:

Line Position	Data Element	Format
1 - 7	Literal Value	SAMPLE:
8	Field Delimiter	Space
9	Instrument[1]	2 positions; alphanumeric
	Field Delimiter	Comma (,)
	Shift	1 position; alpha Code Meaning G Graveyard D Day S Swing
	Field Delimiter	Comma (,)
	Quan Report Type	3 positions; alphanumeric Code Meaning CAL Calibration VER Calibration Verification for extraction methods only EPA EPA Sample BLK Laboratory Blank FBK Field Blank IPR Initial Precision and Accuracy OPR[2] Ongoing Precision and Accuracy
	Field Delimiter	Comma (,)
	Sample Number	5 positions; alphanumeric
	Field Delimiter	Comma (,)
	Bottle Number	2 positions; numeric For EPA Samples - Range: 01-98 Analysis of TCLP Leachate: 99 All others: 00
	Field Delimiter	Comma (,)
	Fraction	1 position; alphanumeric Code Meaning A Acid B Base C Combined acid base/ neutral D Dioxin P Pesticide V Volatile

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[1] **All quantitation files for calibration, precision and recovery, standards and blanks will be tracked by this instrument number within a given laboratory. Changing of this instrument number by the laboratory would necessitate the submittal of new calibration and initial precision and recovery quantitation reports by the laboratory.**

[2] For volatiles, calibration verification and ongoing precision and recovery tests are performed with the OPR report.

Line Position	Data Element	Format
	Field Delimiter	Comma (,)
	Conc/Dilution Factor	11 positions maximum; numeric with colon (:) separating initial and final sample volume NA:NA - used for calibration standards and other runs that are not extracted
	Field Delimiter	Comma (,)
	Date Extracted	MM/DD/YY - X; 10 positions MM - month; 01-12 DD - day; 01-31 YY - year; 83-99 X - shift; G (graveyard) D (day), S (swing) NA - used for calibration standards and other runs that are not extracted
	Dollar Sign ($)	End of data delimiter

Examples - example Sample Lines in headers for volatiles are given below, the examples are ordered to simulate initial and on-going use of the methods. The actual data used in these examples represent synthetic values and cannot be used for data submission. Numbers in brackets ( ) reference paragraph numbers in the methods.

3.1.3.1 Volatile Examples

a. Initial calibration (1624C: 6.7.1, 7.4, 7.5, and 7.6).

(1) For a 10 ug/L initial calibration
SAMPLE: A1,D,CAL,00010,00,V,NA:NA,NA$
(2) For a 20 ug/L initial calibration
SAMPLE: A1,D,CAL,00020,00,V,NA:NA,NA$
(3) For a 50 ug/L initial calibration
SAMPLE: A1,D,CAL,00050,00,V,NA:NA,NA$
(4) For a 100 ug/L initial calibration
SAMPLE: A1,D,CAL,00100,00,V,NA:NA,NA$
(5) For a 200 ug/L initial calibration
SAMPLE: A1,D,CAL,00200,00,V,NA:NA,NA$

b. Initial precision (1624C: 6.7.2, 8.2).

(1) SAMPLE: A1,D,IPR,00020,00,V,NA:NA,NA$
(2) SAMPLE: A1,D,IPR,00020,00,V,NA:NA,NA$

(3) SAMPLE: A1,D,IPR,00020,00,V,NA:NA,NA$

(4) SAMPLE: A1,D,IPR,00020,00,V,NA:NA,NA$

c. Calibration verification (1624C: 6.7.2, 11.5) and on-going precision.

SAMPLE: A1,S,OPR,00020,00,V,NA:NA,NA$

d. Laboratory Blank (1624C: 8.5)

SAMPLE: A1,S,BLK,00000,00,V,1:1,NA$

e. Analysis of samples (typical).

SAMPLE: A1,S,EPA,NNNNN,NN,V,1:1,NA$

SAMPLE: A1,S,EPA,NNNNN,NN,V,1:10,NA$ (is one in 10 dilution of sample above).

f. Analysis of Field Blanks.

(1) With EPA Sample Number

SAMPLE: A1,S,FBK,NNNNN,NN,V,1:1,NA$

(2) Laboratory Composite

SAMPLE: A1,S,FBK,NNNNN,NN,V,1:1,NA$

(where NNNNN represents the 1st sample number of a set for a given day)

3.1.3.2 Semi-volatile Examples

a. Initial calibration (1625C: 6.13, 7.4, 7.5 and 7.6)

(1) For 10 ug/ml initial calibration
SAMPLE: A2,D,CAL,00010,00,C,NA:NA,NA$
(2) For a 20 ug/ml initial calibration
SAMPLE: A2,D,CAL,00020,00,C,NA:NA,NA$
(3) For a 50 ug/ml initial calibrattion
SAMPLE: A2,D,CAL,00050,00,C,NA:NA,NA$
(4) For a 100 ug/ml initial calibration
SAMPLE: A2,D,CAL,00100,00,C,NA:NA,NA$
(5) For a 200 ug/ml initial calibration
SAMPLE: A2,D,CAL,00200,00,C,NA:NA,NA$

b. Initial precision and accuracy (1625C: 8.2).

(1) SAMPLE: A2,D,IPR,00100,00,C,1000:1,MM/DD/YY-G$
(2) SAMPLE: A2,D,IPR,00100,00,C,1000:1,MM/DD/YY-G$
(3) SAMPLE: A2,D,IPR,00100,00,C,1000:1,MM/DD/YY-G$
(4) SAMPLE:
A2,D,IPR,00100,00,C,1000:0.95,MM/DD/YY-G$

c. Calibration verification (1625C: 12.5).

SAMPLE: A2,S,VER,00100,00,C,NA:NA,NA$

d. On-going precision and accuracy (1625C: 12.7).
SAMPLE: A2,S,OPR,00100,00,C,1000:1,MM/DD/YY-D$

e. Laboratory Blank (1625C: 8.5)
SAMPLE: A2,S,BLK,00000,00,C,1000:1,MM/DD/YY-D$

f. Field Blank
SAMPLE: A2,S,FBK,NNNNN,NN,C,1000:1,MM/DD/YY-D$

g. Analysis of samples.

(1) SAMPLE:
A2,S,EPA,NNNNN,NN,B,1000:1,MM/DD/YY-D$
(2) SAMPLE:
A2,S,EPA,NNNNN,NN,A,1000:1,MM/DD/YY-D$
(3) SAMPLE:
A2,S,EPA,NNNNN,NN,A,10:1,MM/DD/YY-D$
(employs the dilute aliquot (1625C: 10.1 and 14.4) for analysis).

(4) SAMPLE:
A2,S,EPA,NNNNN,NN,B,1000:10,MM/DD/YY-D$
(employs dilution of the extract (1625C: 14.4) for analysis).

g. Analysis of Field Blanks.
SAMPLE: A2,S,FBK,NNNNN,NN,C,1000:1,MM/DD/YY-D$

3.1.3.3 Dioxin Examples* (Methods 613 and 613 extended to 10 liter samples)

a. Initial Calibration (613: section 7)

(1) For 200 ng/ml (200 pg/ul, 0.2 ng/ul)
SAMPLE: A2,D,CAL,00200,0,D,NA:NA,NA$
(2) For 1000 ng/ml
SAMPLE: A2,D,CAL,01000,00,D,NA:NA,NA$
(3) For 5000 ng/ml
SAMPLE: A2,D,CAL,05000,00,D,NA:NA,NA$
(4) For 20000 ng/ml
SAMPLE: A2,D,CAL,20000,00,D,NA:NA,NA$
(5) For 40000 ng/ml
SAMPLE: A2,D,CAL,40000,00,D,NA:NA,NA$

---

* Note: Requirements may vary between contracts. Unless specified otherwise in the contract, the compound concentrations given in these examples are to be used.

b. Initial precision and accuracy

- (1) For Method 613 (613: 8.2)
  - (a) SAMPLE:
    A2,D,PAR,01000,00,D,1000:0.05,MM/DD/YY-G$
- (2) For Method 613 extended to 10 liter samples
  - (a) SAMPLE:
    A2,D,PAR,01000,00,D,10000:0.05,MM/DD/YY-G$

c. Calibration Verification (613: 7.1.3)

SAMPLE: A2,D,VER,01000,00,D,NA:NA,NA$

d. Ongoing Precision and Accuracy

- (1) Method 613 (613: Replaces 8.4)
  - (a) SAMPLE:
    A2,D,PAR,01000,00,D,1000:0.05,MM/DD/YY-G$
- (2) Method 613 Extended to 10 liter samples
  - (a) SAMPLE:
    A2,D,PAR,01000,00,D,10000:0.05,MM/DD/YY-G$

e. Laboratory Blank

- (1) Method 613 (613: 8.5)
  - (a) SAMPLE:
    A2,D,BLK,00000,00,D,1000:0.05,MM/DD/YY-G$
- (2) Method 613 Extended to 10 liter samples (613E: 4.3)
  - (a) SAMPLE:
    A2,D,BLK,01000,00,D,10000:0.05,MM/DD/YY-G$

f. Analysis of Samples

- (1) SAMPLE:
  A2,G,EPA,NNNNN,NN,D,1000:0.05,MM/DD/YY-G$
- (2) SAMPLE:
  A2,G,EPA,NNNNN,NN,D,10000:0.05,MM/DD/YY-G$
  (for 10 liter sample)

3.1.4 Condition Line

The Condition line contains the following data elements. As with the sample line, data elements are recorded on the line in the order indicated. Data elements are separated from each other by means of a comma (,). The end of the data element list is signified by a dollar sign ($). The specifications for this type of line are:

Line Position	Data Element	Format
1 - 7	Literal Value	CONDS.:
8	Field Delimiter	Space
9	Method	5 positions; alphanumeric 1624C or 1625C, 613 or 613A, 713
	Field Delimiter	Comma (,)
	Column Length	6 positions; alphanumeric expressed in meters ie. 2.5 M or 35 M; Volatile Range 2.8-3.1 M Semi Volatile Range 25-35 M Dioxin Range 25-65 M
	Field Delimiter	Comma (,)
	Column Inside Diameter	6 positions; alphanumeric expressed in millimeters ie. 2 mm or .3 mm; Volatile Range: 1-3 mm Semi Volatile Range: 0.2 - 0.35 mm Dioxin Range 0.2-0.35 mm
	Field Delimiter	Comma (,)
	Column Initial Temperature	7 positions; numeric an at sign (@) is used to separate Hold and Temperature ie Hold @ Temp Volatile Hold Range: 2-4 min Semi Vol Hold Range: 4-6 min Dioxin Hold Range: 0-10 min Volatile Temp Range: 25-50°C Semi Vol Temp Range: 25-35°C Dioxin Temp Range: 50-200°C
	Field Delimiter	Comma (,)

Line Position	Data Element	Format
	Column Temperature Program	10 positions; numeric with a dash (-) separating initial and final temperatures and with an at sign (@) separating temperature program rate ie. 45-250 @ 8
	Field Delimiter	Comma (,)
	Column Final Temperature	7 positions; numeric with an at sign (@) separating hold and temperature ie. Hold @ Temp Volatile Hold Range: 0-20 min Semi Vol Hold Range: 10-20 min Dioxin Hold Range: 10-30 min Volatile Temp Range: 200-250°C Semi Vol Temp Range: 240-290°C Dioxin Temp Range: 200-300°C
	Field Delimiter	Comma (,)
	Carrier Gas Flow Rate	9 positions; alphanumeric Format: 30ML/M or 30 CM/S; Volatile range: 20-40 ml/min Semi Volatile range: 20-60 cm/sec Dioxin Range: 20-60 cm/sec
	End of Data Delimiter	Dollar sign ($)

3.1.4.1 Volatile Example

CONDS.: 1624C,3.0M,2MM,3@45,45-250@8,1@250,20ML/M$

3.1.4.2 Semi-Volatile Example

CONDS.: 1625C,30M,0.25MM,5@30,30-280@8,15@280,30CM/S$

3.1.4.3 Dioxin Example

CONDS.: 613E,60M,0.25MM,1@150,150-250@12,15@230,30CM/S$

3.2 Compound List Section

The compound list section is the second sic section of information appearing on the quantitation report. It identifi the actual compounds that were determined during analysis. This section is m up of two types of lines:

- Title Line
- Compound Identification Line

3.2.1 Title Line

The title line must appear first withi he Compound List Section. Only one title line may be present in the sec . This line is formatted as follows:

Line Position	Data Element	Format
1	Field Delimiter	Space
2-3	Literal Value	NO
	Field Delimiter	Spaces (At Least 2)
	Literal Value	NAME

3.2.2 Compound Identification Line

A compound identification line is included in this section for each compound that was determined during analysis. Compound lines should be shown in the order in which they were determined. Each compound identification line is made up of three data elements specified in the following order within the line:

- Compound Order Number
- EGD Compound Number
- Compound Name

The compound order number is a numerical code that establishes the order of compound determination by the GC/MS. The code is used on the Quantitation Report to match up compound identification with compound analysis and reference data appearing in subsequent sections of the report. On each quantitation report this number always starts with 1. The number 1 is always assigned to the first compound that is determined, the number 2 to the second compound that is determined and so on.

Table 4 contains a list of valid compound names and EGD compound numbers.

Each compound identification line will appear in the following format:

Line Position	Data Element	Format
1-3	Compound Order Number	1 to 3 character number; right justified in field; range 1-250
4-5	Field Delimiter	Spaces (At Least 2)
	EGD Compound Number	3 positions; numeric Range Meaning 001-129 Quantitated by internal standard 130-199 Misc., internal standard and surrogate compound 201-299 Labeled Compound (isotope) Quantitated by internal standard

Line Position	Data Element	Format	
		Range	**Meaning**
		301-399	Quantitated by isotope dilution
		401-429	Labeled compound (isotope) quantitated by internal standard.
		501-599	Non-priority Pollutant Compounds quantitated by internal standard
		601-699	Synfuel specific and Appendix C labeled compounds (isotopes) quantitated by internal standard
		701-799	Synfuel specific and Appendix C compounds quantitated by isotope dilution.
		800-829	Pollutants 100-129 quantitated by isotope dilution.
		900-999	Non-priority Pollutant Compounds quantitated by internal standard
	Field Delimiter	Spaces (At Least 2)	
	Compound Name	70 positions; alphanumeric	

3.3 Technical Data Section

The technical data section provides measurement data for each compound that is determined. It is the third section within the quantitation report. This section is made up of two types of lines:

- Title Line
- Compound Technical Data Line

3.3.1 Title Line

The title line must appear first within the Technical Data Section. Only one title line may be present in the section. This line is formatted as follows:

Line Position	Data Element	Format
1	Field Delimiter	Space
2-3	Literal Value	NO
	Field Delimiter	Spaces (At Least 2)
	Literal Value	M/E
	Field Delimiter	Spaces (At Least 2)
	Literal Value	SCAN
	Field Delimiter	Spaces (At Least 2)
	Literal Value	TIME
	Field Delimiter	Spaces (At Least 2)
	Literal Value	REF
	Field Delimiter	Spaces (At Least 2)
	Literal Value	RRT
	Free Area	Spaces or other literal values
First non-blank character at or past position 41.	Literal Value	AREA
	Field Delimiter	Spaces (At Least 2)
	Literal Value	AMOUNT
	Free Area	Spaces or other literal values

3.3.2 Compound Technical Data Line

A compound technical data line is included in this section for each compound that is determined. Compound technical data lines are ordered the same as the compound identification lines in the compound list section. The compound order number is used for this purpose and serves to connect compound identification with the technical data. The compound technical data line at a minimum must contain the following data elements.

- Compound Order Number
- Mass to Charge Ratio
- Scan Number
- Retention Time
- Reference Compound
- Relative Retention Time
- Peak Area
- Amount
- Unit of Measure

The specific format for this line is as follows:

Line Position	Data Element	Format
1-3	Compound Order	3 positions; numeric; right-Numberjustified; Range 1-250
	Field Delimiter	Spaces (At Least 1)
	Mass to Charge Ratio (M/Z)	4 positions; numeric; Volatile range: 20-250; Semi Volatile range: 35-450 Dioxin (Selected Ion Monitoring): 257, 320, 322, 323, 328, 332, and 334.
	Field Delimiter	Spaces (At Least 1)
	Scan Number	5 positions; numeric; range 1-9999
	Field Delimiter	Spaces (At Least 1)
	Retention Time	6 positions; numeric with colon; format; MM:SS
	Field Delimiter	Spaces (At Least 1)
	Reference Compound	3 positions; numeric; range 1-250

Line Position	Data Element	Format
	Field Delimiter	Spaces (At Least 1)
	Relative Retention Time	5 positions; numeric with decimal point and 3 decimal places
	Field Delimiter	Spaces (At Least 1)
	Open Field	Spaces or other field value
First non-blank character at or past position 41.	Peak Area	10 positions; numeric
	Field Delimiter	Spaces (At Least 1)
	Amount	10 positions; numeric with decimal point and 3 decimal places
	Field Delimiter	Spaces (At Least 1)
	Unit of Measure	5 positions; alphanumeric Valid codes: uG/L, uG/ml, or ng/ml
	Field Delimiter	Spaces (At Least 1)
	Open Area	Spaces or other field values.

3.4 Reference Data Section

The reference data section is the fourth section that must appear on each Quantitation Report. It provides reference and library data about the analysis that was performed. It is made up of two types of lines:

- Title Line
- Compound Reference Data Line

3.4.1 Title Line

The title line must appear first within this section. Only one title line is permitted within the section. Subsequent title lines will be deleted if present. The line is formatted as follows:

Line Position	Data Element	Format
1	Field Delimiter	Space
2-3	Literal Value	NO
	Field Delimiter	Spaces (At Least 2)
	Literal Value	RET (L)
	Field Delimiter	Spaces (At Least 1)
	Open Area	Spaces or other literal values
First non-blank character at or past position 19.		Spaces (At Least 1)
	Literal Value	RRT (L)
	Field Delimiter	Spaces (At Least 1)
	Open Area	Spaces or other literal values
First non-blank character at or past position 43.	Literal Value	AMNT (L)
	Field Delimiter	Spaces (At Least 1)
	Literal Value	R.FAC
	Field Delimiter	Spaces (At Least 1)
	Literal Value	R.FAC (L)
	Open Area	Spaces or other literal values

3.4.2 Compound Reference Data Line

A compound reference data line is included in this section for each compound that is determined. These lines are ordered the same as the compound identification lines in the compound list section. The compound order number is used for this purpose and serves to connect the compound identification with the reference data. This means that there is a one to one correspondence between the compound identification lines and the reference data lines. The reference data line at a minimum must contain the following data elements.

- Compound Order Number
- Library Retention Time
- Library Relative Retention Time
- Library Amount
- Response Factor
- Library Response Factor

The specific format for this line is as follows:

Line Position	Data Element	Format
1-3	Compound Order Number	3 positions; numeric; Range 1-250
	Field Delimiter	Spaces (At Least 2)
	Library Retention Time	6 positions; numeric with colon; format - MM:SS
	Field Delimiter	Spaces (At Least 1)
	Open Area	Spaces or other data values
First non-blank character starting at or past position 18.	Library Relative Retention Time	5 positions

Line Position	Data Element	Format
	Field Delimiter	Spaces (At Least 1)
	Open	Spaces or other data values
First non-blank character starting at or past position 41.	Library Amount	9 positions; numeric with decimal point and 2 decimal places
	Field Delimiter	Spaces (At Least 1)
	Response Factor	7 positions; numeric with decimal point and 3 decimal places
	Field Delimiter	Spaces (At Least 1)
	Library Response Factor	7 positions; numeric with decimal point and 3 decimal places.
	Field Delimiter	Spaces (At Least 1)
	Open Area	Spaces or other data values.

3.5 Associated Quantitation Reports

Some GCMS data systems will not permit all data from an analysis to be given in a single quantitation report when the number of compounds analyzed exceeds a certain limit (cases reported to date are 80, 110, 125 entries). Because the semi-volatiles' analysis requires data for more than 125 entries, only the semi-volatiles' quantitation report may be divided into more than one report. These multiple quantitation reports are called "Associated quantitation reports." In these reports, the header shall be identical, except that a different name may appear after the word "FILE" (see, e.g., the associated quantitation reports given by example in Table 1B). If associated quantitation reports are submitted, data for all compounds except the 2,2'-difluorobiphenyl internal standard shall appear only once in the totality of the associated quantitation reports. The data for the 2,2'-difluorobiphenyl internal standard shall be identical on each of the associated quantitation reports, except for the data in the "%TOT" column. No more than three associated quantitation reports are permitted for any analysis.

3.6 Non-alphanumeric Characters

Sections 3.1 through 3.4 define the character type (alphabetical or numeric) permitted in each field on the quantitation report. Other characters are prohibited. Specifically, the minus sign (-) and the asterisk (*) are not permitted in any field in the Technical Data Section (Section 3.3) or any Reference Data Section (Section 3.4). Negative numbers are prohibited because they have no meaning in quantitation or retention time; asterisks are prohibited because they indicate an overflow condition in most FORTRAN language systems, and are usually the result of an attempt to divide by zero. If the amount for a given compound is negative, and the amount is to be interpreted as "not found," the quantitation report shall conform to the "Reporting of Compounds Not Found" in Section 4.b. If an overflow condition exists, and is the result of an attempt to divide by zero, an alternate means of reporting shall be used which provides an accurate estimate of the amount for the compound.

4. **Reporting of Quantitation Data**

a. Unit of Work

Each tape submitted must be a complete unit of work, processable within itself.

1) Each tape submitted must begin with either a calibration (CAL) or calibration verification (OPR for volatiles, VER for semi-volatiles) run.

2) All quantitation reports from a single shift must be reported on the same tape.

3) All associated calibration (CAL) runs and initial precision and recovery (IPR) runs must be on the same tape.

4) The number of quantitation reports contained on a single tape must not exceed 300.

b. Reporting of Compounds Not Found

Compounds not appearing within a quantitation report (e.g. all data lines for a compound have been deleted from the report) are assumed to be not found. If a not found compound is reported within the body of the quantitation report the following specifications are to be followed for reporting of these compounds.

1) The Compound Identification Line must be complete.

2) The Compound Technical Data Line must be reported in one of the following formats:

a) Compound order number, 1-3 spaces, and the literal value 'NOT FOUND'. All other data elements in the line must be blank or space filled on the data line.

b) Compound Order Number, the Mass to Charge Ratio (M/Z) and the Reference Compound. All other data elements must be blank or space filled on the data line.

3) The Compound Reference Data Line must contain, at a minimum, only the Compound Order Number.

c. Alternate Reporting for Problem Compounds

If an interference or other problem precludes automated quantitation by the GCMS software, Report Form I (attached as Table 3) shall be used and shall supercede data on magnetic tape, the "note" column on this form shall be used to reference a comment which describes the inteference or problem, and Report Form I shall be submitted with data for the sample to which it applies.

5. Packaging and Shipping Instructions

Packaging Requirements:

- Each tape reel shall bear a tape label containing the laboratory tape ID, the laboratory name, the tape density, the block size, and the number of files.

- Each tape package shall contain in addition to the tape reel(s) at least one Quantitation Report Magnetic Tape Transmittal Form for each tape reel (see Table 2 for a sample transmittal form and transmittal form description), all Lab Chronicle Reports associated with the reported samples, and BFB or DFTPP spectra analysis required per shift per machine.

- Tape reels with their associated transmittal forms and chronicle reports shall be packaged in such a way to ensure their safety and integrity. The outside of all packages should be marked with a 'DO NOT X-RAY' label. It will be the laboratory's responsibility to replace any tape, quantitation report data, and accompanying documents damaged during shipping to the Sample Control Center.

Shipping Requirements:

Tape packages shall be shipped either by the U.S. Postal Service or by any carrier with direct delivery.

Shipping address by U.S. Postal Service:

Industrial Technology Division
Sample Control Center
P.O. Box 1407
Alexandria, VA 22313

Shipping address by other carriers:

Industrial Technology Division
Sample Control Center
Suite 200
300 N. Lee St.
Alexandria, VA 22314

TABLE 1A EXAMPLE OF VOLATILE QUANTITATION REPORT

```
QUANTITATION REPORT    FILE: 7VMIX200

DATA: 7VMIX200.TI
07/07/86 22:24:00
SAMPLE: S1,S,CAL,00200,00,V,NA:NA,NA*
CONDS.: 1624B,

AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)
RESP. FAC. FROM AVERAGE OF WHOLE .RL

 NO  NAME
  1  181 IS**BROMOCHLOROMETHANE**
  2  245 (D3) 45V METHYL CHLORIDE
  3  345 45V METHYL CHLORIDE
  4  246 (D3)46V METHYL BROMIDE
  5  346 46V METHYL BROMIDE
  6  288 (D3)88V VINYL CHLORIDE
  7  388 88V VINYL CHLORIDE
  8  216 (D5) 16V CHLOROETHANE
  9  316 16V CHLOROETHANE
 10  244 (D2) 44V METHYLENE CHLORIDE
 11  344 44V METHYLENE CHLORIDE
 12  616 (D6) ACETONE
 13  516 ACETONE
 14  202 (D2)2V ACROLEIN
 15  002 ACROLEIN
 16  203 (D3)3V ACRYLONITRILE
 17  303 3V ACRYLONITRILE
 18  229 (D2)29V 1,1-DICHLOROETHYLENE
 19  329 29V 1,1-DICHLOROETHYLENE
 20  213 (D3)1,1-DICHLOROETHANE
 21  313 13V 1,1-DICHLOROETHANE
 22  230 (D2)TRANS-1,2-DICHLOROETHYLENE
 23  330 TRANS-1,2-DICHLOROETHYLENE
 24  615 (D10) DIETHYL ETHER
 25  715 DIETHYL ETHER
 26  223 (C13) 23V CHLOROFORM
 27  323 23V CHLOROFORM
 28  210 (D4)1,2-DICHLOROETHANE
 29  310 10V 1,2-DICHLOROETHANE
 30  614 (D3) MEK
 31  514 METHYL ETHYL KETONE
 32  211 (D3)11V 1,1,1-TRICHLOROETHANE
 33  311 11V 1,1,1-TRICHLOROETHANE
 34  627 (D8) 1,4-DIOXANE
 35  527 1,4-DIOXANE
 36  182 IS ***2-BROMO-1-CHLOROPROPANE***
 37  206 (13C)CARBONTETRACHLORIDE
 38  306 6V CARBONTETRACHLORIDE
 39  248 (13C)48V DICHLOROBROMOMETHANE
 40  348 48V DICHLOROBROMOMETHANE
 41  232 (D6)32V 1,2-DICHLOROPROPANE
 42  332 32V 1,2-DICHLOROPROPANE
 43  233 (D4)33V TRANS 1,3-DICHLOROPROPYLENE
 44  333 33V TRANS-1,3-DICHLOROPROPYLENE
 45  287 (13C2)87V TRICHLOROETHYLENE
 46  387 87V TRICHLOROETHYLENE
 47  204 (D6)4V BENZENE
 48  304 4V BENZENE
 49  251 (13C)51V CHLORODIBROMOMETHANE
```

TABLE 1A EXAMPLE OF VOLATILES QUANTITATION REPORT

```
50  351 51V CHLORODIBROMOMETHANE
51  214 (13C2)14V 1,1,2-TRICHLOROETHANE
52  314 14V 1,1,2-TRICHLOROETHANE
53  019 19V 2-CHLOROETHYL VINYL ETHER
54  247 (13C) 47V BROMOFORM
55  047 47V BROMOFORM
56  183 IS ***1,4-DICHLOROBUTANE***
57  215 (D2) 15V 1,1,2,2-TETRACHLOROETHANE
58  315 15V 1,1,2,2-TETRACHLOROETHANE
59  285 (13C2) 85V TETRACHLOROETHYLENE
60  085 85V TETRACHLOROETHYLENE
61  286 (D5) TOLUENE
62  386 86V TOLUENE
63  207 (D5) 7V CHLOROBENZENE
64  307 7V CHLOROBENZENE
65  238 (D10) 38V ETHYLBENZENE
66  338 38V ETHYLBENZENE
67  185 BFB BROMOFLUOROBENZENE

NO  M/E  SCAN   TIME  REF   RRT   METH     AREA(HGHT)  AMOUNT            %TOT
 1  128   251  12:46    1  1.000  A BB        45612.     20.000 UG/L     0.17
 2   53    60   3:03    1  0.239  A?BB       161108.     19.258 UG/L     0.17
 3   50    60   3:03    2  1.000  RXRY      1797960.    200.559 UG/L     1.72
 4   99    93   4:44    1  0.371  A BB       102000.     21.427 UG/L     0.18
 5   96    94   4:47    4  1.011  RXRY      1441280.    199.944 UG/L     1.71
 6   65   115   5:51    1  0.458  A BB       200409.     21.743 UG/L     0.19
 7   62   117   5:57    6  1.017  RXRY      2102360.    203.499 UG/L     1.74
 8   71   140   7:07    1  0.558  A BB        37562.     23.147 UG/L     0.20
 9   64   143   7:16    8  1.021  A BB      1057170.    201.312 UG/L     1.73
10   88   184   9:21    1  0.733  A?BB       178840.     32.976 UG/L     0.28
11   84   185   9:24   10  1.005  RXRY      2904630.    203.212 UG/L     1.74
12  NOT  FOUND
13   58   204  10:22    1  0.813  QEDT       257295.    944.069 UG/L     8.09
14  NOT  FOUND
15   56   205  10:25    1  0.817  QEDT       472521.   1007.150 UG/L     8.63
16   56   212  10:47    1  0.845  QEDT       106044.     37.106 UG/L     0.32
17   53   216  10:59   16  1.019  RXRY       962244.    453.130 UG/L     3.88
18   65   248  12:36    1  0.988  A BB        41974.     20.878 UG/L     0.18
19   61   249  12:39   18  1.004  RXRY      1194350.    203.878 UG/L     1.75
20   66   272  13:50    1  1.084  A BB       170999.     21.431 UG/L     0.18
21   63   273  13:53   20  1.004  RXRY      1828310.    205.744 UG/L     1.76
22   65   289  14:41    1  1.151  QEDT        50772.     22.747 UG/L     0.19
23   61   291  14:48   22  1.007  RXRY       974562.    187.085 UG/L     1.60
24   84   296  15:03    1  1.179  A BB       272891.     24.933 UG/L     0.21
25   74   301  15:18   24  1.017  RXRY      3928690.    982.383 UG/L     8.42
26   86   296  15:03    1  1.179  A BB       112008.     23.337 UG/L     0.20
27   85   296  15:03   26  1.000  RXRY      1191490.    203.036 UG/L     1.74
28   67   313  15:55    1  1.247  A BB        39392.     20.254 UG/L     0.17
29   62   315  16:01   28  1.006  RXRY       957344.    201.271 UG/L     1.72
30  NOT  FOUND
31   72   317  16:07    1  1.263  A BB       339211.   1010.870 UG/L     8.66
32  102   344  17:29    1  1.371  A BB        85173.     20.481 UG/L     0.18
33   97   345  17:32   32  1.003  RXRY      1276290.    206.138 UG/L     1.77
34  NOT  FOUND
35   88   343  17:26    1  1.367  A BB        23247.   1278.140 UG/L    10.95
36   77   456  23:11   36  1.000  A?BB       163747.     20.000 UG/L     0.17
37   48   356  18:06   36  0.781  QEDT        28437.     29.538 UG/L     0.25
38   47   355  18:03   37  0.997  RXRY       255185.    182.656 UG/L     1.57
39   86   359  18:15   36  0.787  A BB       123738.     25.016 UG/L     0.21
40   83   359  18:15   39  1.000  RXRY      1505850.    167.841 UG/L     1.44
41   67   393  19:59   36  0.862  A BB       1[illegible]0.     20.708 UG/L     0.18
```

TABLE 1A   EXAMPLE OF VOLATILES QUANTITATION REPORT

```
42   63   396  20:08  41  1.008  RXRY   1068560.    210.283 UG/L   1.80
43   79   396  20:08  36  0.868  A?BB    111843.     28.327 UG/L   0.24
44   75   399  20:17  43  1.008  RXRY   2832010.    193.967 UG/L   1.66
45  136   412  20:57  36  0.904  A BB     63685.     30.408 UG/L   0.26
46   95   412  20:57  45  1.000  RXRY   1997970.    193.946 UG/L   1.66
47   84   426  21:39  36  0.934  A BB    385811.     21.912 UG/L   0.19
48   78   429  21:48  47  1.007  RXRY   2979110.    192.241 UG/L   1.65
49  130   419  21:18  36  0.919  A?VB    109992.     26.522 UG/L   0.23
50  129   420  21:21  49  1.002  RXRY    686162.    161.048 UG/L   1.38
51   84   426  21:39  36  0.934  A BB    385811.     21.912 UG/L   0.19
52   83   425  21:36  51  0.998  RXRY    730003.    190.113 UG/L   1.63
53  106   453  23:02  36  0.993  A BB    139858.    212.542 UG/L   1.82
54  176   479  24:21  36  1.050  A BB      5954.     42.778 UG/L   0.37
55  173   479  24:21  36  1.050  A BB    124971.    299.567 UG/L   2.57
56   55   544  27:39  56  1.000  A?BB    119766.     20.000 UG/L   0.17
57   84   530  26:56  56  0.974  A?BB     95140.     21.419 UG/L   0.18
58   83   531  27:00  57  1.002  RXRY    976982.    213.446 UG/L   1.83
59  172   537  27:18  56  0.987  A BB     18666.     30.772 UG/L   0.26
60  164   537  27:18  56  0.987  A BB    831510.    220.038 UG/L   1.89
61   97   570  28:58  56  1.048  A BB    172719.     20.224 UG/L   0.17
62   92   573  29:08  61  1.005  RXRY   1806000.    202.499 UG/L   1.74
63  117   597  30:21  56  1.097  A?BB    191790.     20.124 UG/L   0.17
64  112   600  30:30  63  1.005  RXRY   2106290.    203.388 UG/L   1.74
65  116   652  33:09  56  1.199  A BB     93772.     19.921 UG/L   0.17
66  106   659  33:30  65  1.011  RXRY    843200.    205.244 UG/L   1.76
67  NOT FOUND

NO   RET(L) RATIO RRT(L) RATIO     AMNT    AMNT(L)    R.FAC R.FAC(L) RATIO
 1   12:42  1.00  1.000  1.00     20.00     20.00    1.000   1.000    1.00
 2    3:03  1.00  0.238  1.00     19.26     20.00    3.532   3.668    0.96
 3    3:06  0.98  0.242  4.13    200.56     20.00   11.160   1.113   10.03
 4    4:47  0.99  0.373  0.99     21.43     20.00    2.236   2.087    1.07
 5    4:53  0.98  0.381  2.65    199.94     20.00   14.130   1.413   10.00
 6    5:54  0.99  0.460  1.00     21.74     20.00    4.394   4.042    1.09
 7    6:00  0.99  0.468  2.17    203.50     20.00   10.490   1.031   10.17
 8    7:10  0.99  0.560  1.00     23.15     20.00    0.824   0.712    1.16
 9    7:19  0.99  0.571  1.79    201.31     20.00   28.145   2.796   10.07
10    9:24  0.99  0.734  1.00     32.98     20.00    3.921   2.378    1.65
11    9:27  0.99  0.738  1.36    203.21     20.00   16.241   1.598   10.16
12   10:19        0.806                     20.00            1.000
13   10:25  1.00  0.813  1.00    944.07    100.00    1.128   0.120    9.44
14   10:34        0.825                     20.00            1.000
15   10:34  0.99  0.825  0.99   1007.15    100.00    2.072   0.206   10.07
16   11:02  0.98  0.861  0.98     37.11     20.00    2.325   1.253    1.86
17   11:08  0.99  0.869  1.17    453.13    100.00    1.815   0.401    4.53
18   12:36  1.00  0.984  1.00     20.88     20.00    0.920   0.882    1.04
19   12:39  1.00  0.988  1.02    203.88     20.00   28.455   2.791   10.19
20   13:50  1.00  1.079  1.00     21.43     20.00    3.749   3.499    1.07
21   13:53  1.00  1.083  0.93    205.74     20.00   10.692   1.039   10.29
22   14:32  1.01  1.135  1.01     22.75     20.00    1.113   0.979    1.14
23   14:48  1.00  1.155  0.87    187.08     20.00   19.195   2.052    9.35
24   15:03  1.00  1.175  1.00     24.93     20.00    5.983   4.799    1.25
25   15:18  1.00  1.194  0.85    982.38    100.00    2.879   0.293    9.82
26   15:03  1.00  1.175  1.00     23.34     20.00    2.456   2.104    1.17
27   15:03  1.00  1.175  0.85    203.04     20.00   10.638   1.048   10.15
28   15:55  1.00  1.242  1.00     20.25     20.00    0.864   0.853    1.01
29   16:04  1.00  1.254  0.80    201.27     20.00   24.303   2.415   10.06
30   15:45        1.230                     20.00            1.000
31   16:07  1.00  1.258  1.00   1010.87    100.00    1.487   0.147   10.11
32   17:29  1.00  1.365  1.00     20.48     20.00    1.867   1.824    1.02
33   17:35  1.00  1.373  0.73    206.14     20.00   14.985   1.454   10.31
```

TABLE 1A EXAMPLE OF VOLATILES QUANTITATION REPORT

34	18:30		1.357			20.00		1.000	
35	17:23	1.00	1.357	1.01	1278.15	100.00	0.102	0.008	12.78
36	23:11	1.00	1.000	1.00	20.00	20.00	1.000	1.000	1.00
37	18:06	1.00	0.779	1.00	29.54	20.00	0.174	0.118	1.48
38	18:03	1.00	0.779	1.28	182.66	20.00	8.974	0.983	9.13
39	18:15	1.00	0.787	1.00	25.02	20.00	0.756	0.604	1.25
40	18:15	1.00	0.787	1.27	167.84	20.00	12.170	1.450	8.39
41	19:59	1.00	0.862	1.00	20.71	20.00	0.643	0.621	1.04
42	20:08	1.00	0.862	1.17	210.28	20.00	10.153	0.966	10.51
43	20:11	1.00	0.871	1.00	28.33	20.00	0.683	0.482	1.42
44	20:17	1.00	0.875	1.15	193.97	20.00	25.321	2.611	9.70
45	20:57	1.00	0.904	1.00	30.41	20.00	0.389	0.256	1.52
46	20:57	1.00	0.904	1.11	193.95	20.00	31.373	3.235	9.70
47	21:39	1.00	0.941	0.99	21.91	20.00	2.356	2.151	1.10
48	21:48	1.00	0.941	1.07	192.24	20.00	7.722	0.803	9.61
49	21:21	1.00	0.921	1.00	26.52	20.00	0.672	0.507	1.33
50	21:21	1.00	0.921	1.09	161.05	20.00	6.238	0.775	8.05
51	21:39	1.00	0.934	1.00	21.91	20.00	2.356	2.151	1.10
52	21:36	1.00	0.932	1.07	190.11	20.00	1.892	0.199	9.51
53	23:02	1.00	0.993	1.00	212.54	20.00	0.854	0.080	10.63
54	24:21	1.00	1.050	1.00	42.78	20.00	0.036	0.017	2.14
55	24:21	1.00	1.050	1.00	299.57	20.00	0.763	0.051	14.98
56	27:36	1.00	1.000	1.00	20.00	20.00	1.000	1.000	1.00
57	26:56	1.00	0.976	1.00	21.42	20.00	0.794	0.742	1.07
58	27:00	1.00	0.978	1.02	213.45	20.00	10.269	0.962	10.67
59	27:18	1.00	0.989	1.00	30.77	20.00	0.156	0.101	1.54
60	27:18	1.00	0.989	1.00	220.04	20.00	6.943	0.631	11.00
61	28:58	1.00	1.050	1.00	20.22	20.00	1.442	1.426	1.01
62	29:08	1.00	1.055	0.95	202.50	20.00	10.456	1.033	10.12
63	30:21	1.00	1.099	1.00	20.12	20.00	1.601	1.592	1.01
64	30:30	1.00	1.105	0.91	203.39	20.00	10.982	1.080	10.17
65	33:09	1.00	1.201	1.00	19.92	20.00	0.783	0.786	1.00
66	33:30	1.00	1.214	0.83	205.24	20.00	8.992	0.876	10.26
67	27:24		1.342			10.00		1.000	

TABLE 1B EXAMPLE OF SEMIVOLATILES QUANTITATION REPORT

```
QUANTITATION REPORT    FILE: OPR808

DATA: OPR808.TI
08/25/86 17:52:00
SAMPLE: S3,S,OPR,00100,00,C,1000:1,08/08/86-D$
CONDS.: 1625B,

AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)
RESP. FAC. FROM AVERAGE OF WHOLE .RL

 NO  NAME
  1  164 2,2'-DIFLUOROBIPHENYL
  2  201 (D10)ACENAPHTHENE
  3  301 ACENAPHTHENE
  4  277 (D8)ACENAPHTHYLENE
  5  377 ACENAPHTHYLENE
  6  281 (D10)PHENANTHRENE
  7  381 PHENANTHRENE
  8  278 (D10)ANTHRACENE
  9  378 ANTHRACENE
 10  205 (D8)BENZIDENE
 11  305 BENZIDENE
 12  272 (D12)BENZO(A)ANTHRACENE
 13  372 BENZO(A)ANTHRACENE
 14  276 (D12)CHRYSENE
 15  376 CHRYSENE
 16  274 (D12)BENZO(B)FLUORANTHENE
 17  374 BENZO(B)FLUORANTHENE
 18  275 (D12)BENZO(K)FLUORANTHENE
 19  375 BENZO(K)FLUORANTHENE
 20  273 (D12)BENZO(A)PYRENE
 21  373 BENZO(A)PYRENE
 22  279 (D12)BENZO(G,H,I)PERYLENE
 23  379 BENZO(G,H,I)PERYLENE
 24  083 INDENOPYRENE
 25  082 DIBENZO(A,H)ANTHRACENE
 26  612 (D10) BIPHENYL
 27  712 BIPHENYL
 28  218 (D8) BIS(2-CHLOROETHYL)ETHER
 29  318 BIS(2-CHLOROETHYL)ETHER
 30  043 BIS(2-CHLOROETHOXY)METHANE
 31  242 (D12)BIS(2-CHLOROISOPROPYL)ETHER
 32  342 BIS(2-CHLOROISOPROPYL)ETHER
 33  266 (D4)BIS(2-ETHYLHEXYL)PHTHALATE
 34  366 BIS(2-ETHYLHEXYL)PHTHALATE
 35  041 4-BROMOPHENYLPHENYLETHER
 36  067 BUTYLBENZYLPHTHALATE
 37  617 (D22)N-DECANE
 38  717 N-DECANE
 39  606 (D26)N-DODECANE
 40  706 N-DODECANE
 41  619 (D34)N-HEXADECANE
 42  719 N-HEXADECANE
 43  621 (D42)N-EICOSANE
 44  721 N-EICOSANE
 45  623 (D50)N-TETRACOSANE
 46  723 N-TETRACOSANE
 47  626 (D62)N-TRIACONTANE
 48  726 N-TRICONTANE
 49  220 (D7)2-CHLORONAPHTHALENE
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TABLE 1B EXAMPLE OF SEMIVOLATILES QUANTITATION REPORT

```
50   320 2-CHLORONAPHTHALENE
51   240 (D5)4-CHLOROPHENYLPHENYLETHER
52   340 4-CHLOROPHENYLPHENYLETHER
53   613 (D14)P-CYMENE
54   713 P-CYMENE
55   605 (D8)DIBENZOFURAN
56   705 DIBENZOFURAN
57   604 (D8)DIBENZOTHIOPHENE
58   704 DIBENZOTHIOPHENE
59   268 (D4)DI-N-BUTYLPHTHALATE
60   368 DI-N-BUTYLPHTHALATE
61   226 (D4)1,3 DICHLOROBENZENE
62   326 1,3 DICHLOROBENZENE
63   227 (D4)1,4 DICHLOROBENZENE
64   327 1,4 DICHLOROBENZENE
65   225 (D4)1,2 DICHLOROBENZENE
66   325 1,2 DICHLOROBENZENE
67   228 (D8) 3,3' DICHLOROBENZIDINE
68   328 3,3'DICHLOROBENZIDINE
69   270 (D4)DIETHYLPHTHALATE
70   370 DIETHYLPHTHALATE
71   271 (D4)DIMETHYLPHTHALATE
72   371 DIMETHYLPHTHALATE
73   235 (D3)2,4 DINITROTOLUENE
74   335 2,4 DINITROTOLUENE
75   236 (D3)2,6 DINITROTOLUENE
76   336 2,6 DINITROTOLUENE

NO   M/E  SCAN   TIME  REF   RRT  METH     AREA(HGHT)  AMOUNT          %TOT
 1   190  1060  18:33   1  1.000  A BB     107460.     100.000 UG/ML  1.08
 2   164  1184  20:43   1  1.117  QEDT      70129.      91.860 UG/ML  0.99
 3   154  1190  20:49   2  1.005  A BB     123654.     141.982 UG/ML  1.54
 4   160  1153  20:11   1  1.088  A BB      50064.      73.990 UG/ML  0.80
 5   152  1156  20:14   4  1.003  A BB     186728.     176.889 UG/ML  1.91
 6   188  1443  25:15   1  1.361  A?BV     110518.      92.384 UG/ML  1.00
 7   178  1447  25:19   6  1.003  QEDT     185306.     144.103 UG/ML  1.56
 8   188  1452  25:25   1  1.370  A?VB     105243.      97.668 UG/ML  1.06
 9   178  1455  25:28   8  1.002  A?BB     175722.     136.884 UG/ML  1.48
10   192  1698  29:43   1  1.602  QEDT      10270.      55.499 UG/ML  0.60
11   184  1700  29:45  10  1.001  RXRY      14501.     115.217 UG/ML  1.25
12   240  1913  33:29   1  1.805  A?BV      98172.     115.999 UG/ML  1.26
13   228  1918  33:34  12  1.003  QEDT     171666.     135.839 UG/ML  1.47
14   240  1921  33:37   1  1.812  A?VB      93726.     129.095 UG/ML  1.40
15   228  1926  33:42  14  1.003  A BB     169461.     133.483 UG/ML  1.44
16   264  2100  36:45   1  1.981  QEDT      90405.     120.360 UG/ML  1.30
17   252  2105  36:50  16  1.002  RXRY     146838.     119.497 UG/ML  1.29
18   264  2105  36:50   1  1.986  QEDT     105743.     122.839 UG/ML  1.33
19   252  2108  36:53  18  1.001  RXRY     154233.     129.900 UG/ML  1.41
20   264  2151  37:39   1  2.029  A BB      89381.     142.435 UG/ML  1.54
21   252  2158  37:46  20  1.003  RXRY     142419.     125.263 UG/ML  1.36
22   288  2470  43:13   1  2.330  A BB      60675.     143.628 UG/ML  1.55
23   276  2485  43:29  22  1.006  RXRY     118967.     127.058 UG/ML  1.37
24   276  2412  42:13   1  2.275  QEDT      80395.     129.628 UG/ML  1.40
25   278  2417  42:18   1  2.280  QEDT      19622.     195.056 UG/ML  2.11
26   164  1084  18:58   1  1.023  A BB     132581.     101.468 UG/ML  1.10
27   154  1087  19:01  26  1.003  RXRY     297557.     152.349 UG/ML  1.65
28   101   623  10:54   1  0.588  A BB      12473.      80.978 UG/ML  0.88
29    93   632  11:04  28  1.014  A?BB      79499.     143.827 UG/ML  1.56
30    93   853  14:56   1  0.805  A BB      84974.     125.253 UG/ML  1.36
31   131   712  12:28   1  0.672  QEDT      18621.      98.662 UG/ML  1.07
32   121   722  12:38  31  1.014  A BB      22981.     129.493 UG/ML  1.40
```

TABLE 1B EXAMPLE OF SEMIVOLATILES QUANTITATION REPORT

```
33 [illegible]  1960  34:18   1  1.849  A BB      83029.    117.971 UG/ML  1.28
34 [illegible]  1962  34:20  33  1.001  A BB     133048.    138.838 UG/ML  1.50
35  248  1371  24:00   1  1.293  A?BB      36900.    138.603 UG/ML  1.50
36  149  1845  32:17   1  1.741  A BB      88137.    161.249 UG/ML  1.74
37   66   631  11:03   1  0.595  A?VV      29963.     30.748 UG/ML  0.33
38   55   651  11:24  37  1.032  RXRY      14490.    218.974 UG/ML  2.37
39   66   878  15:22   1  0.828  QEDT      46577.     51.076 UG/ML  0.55
40   55   898  15:43  39  1.023  RXRY      17707.    210.159 UG/ML  2.27
41   66  1268  22:11   1  1.196  A BB      70158.     89.423 UG/ML  0.97
42   55  1290  22:34  41  1.017  RXRY      25524.    142.746 UG/ML  1.54
43   66  1581  27:40   1  1.492  A BB      61077.     92.696 UG/ML  1.00
44   55  1606  28:06  43  1.016  RXRY      30946.    153.675 UG/ML  1.66
45   66  1845  32:17   1  1.741  QEDT      59993.     97.947 UG/ML  1.06
46   55  1872  32:46  45  1.015  QEDT      25671.    135.639 UG/ML  1.47
47   66  2187  38:16   1  2.063  A BB      57395.    107.938 UG/ML  1.17
48   55  2227  38:58  47  1.018  RXRY      36742.    167.273 UG/ML  1.81
49  169  1081  18:55   1  1.020  A BB      77165.     89.813 UG/ML  0.97
50  162  1084  18:58  49  1.003  RXRY      92149.    138.562 UG/ML  1.50
51  209  1285  22:29   1  1.212  A BB      37993.     86.243 UG/ML  0.93
52  204  1288  22:32  51  1.002  A?BB      65993.    147.660 UG/ML  1.60
53 [illegible]   669  11:42   1  0.631  QEDT      70927.     68.837 UG/ML  0.74
54 [illegible]   681  11:55  53  1.018  A BB      96523.    117.571 UG/ML  1.27
55  176  1215  21:16   1  1.146  A?BB     109759.     89.708 UG/ML  0.97
56  168  1219  21:20  55  1.003  QEDT     164942.    135.998 UG/ML  1.47
57  192  1422  24:53   1  1.342  A?BB     207620.    180.495 UG/ML  1.95
58  184  1425  24:56  57  1.002  QEDT     164667.    150.116 UG/ML  1.62
59  153  1579  27:38   1  1.490  A BB     131129.     99.932 UG/ML  1.08
60  149  1581  27:40  59  1.001  RXRY     201667.    136.684 UG/ML  1.48
61  152   647  11:19   1  0.610  A?BB      13868.     41.343 UG/ML  0.45
62  146   650  11:22  61  1.005  A?BV      48563.    202.809 UG/ML  2.19
63  152   657  11:30   1  0.620  QEDT      16980.     47.511 UG/ML  0.51
64  146   660  11:33  63  1.005  A BB      56032.    183.677 UG/ML  1.99
65  152   688  12:02   1  0.649  QEDT      20105.     59.045 UG/ML  0.64
66  146   660  11:33  65  0.959  QEDT      51042.    151.967 UG/ML  1.64
67  258  1923  33:39   1  1.814  A?BB      28439.    106.412 UG/ML  1.15
68  252  1924  33:40  67  1.001  RXRY      31307.     96.514 UG/ML  1.04
69  153  1288  22:32   1  1.215  QEDT      47300.     57.280 UG/ML  0.62
70  149  1290  22:34  69  1.002  QEDT      99372.    182.594 UG/ML  1.98
71  167  1159  20:17   1  1.093  QEDT      48416.     47.168 UG/ML  0.51
72  163  1160  20:18  71  1.001  A BB      87639.    172.882 UG/ML  1.87
73  168  1237  21:39   1  1.167  QEDT      27625.     94.980 UG/ML  1.03
74  165  1239  21:41  73  1.002  QEDT      44185.    144.654 UG/ML  1.57
75  167  1166  20:24   1  1.100  A BB      21713.     96.545 UG/ML  1.04
76  165  1169  20:27  75  1.003  A?VB      32491.    130.179 UG/ML  1.41

NO  RET(L)  RATIO  RRT(L)  RATIO    AMNT    AMNT(L)    R.FAC  R.FAC(L)  RATIO
 1  18:38   1.00   1.000   1.00   100.00    100.00    1.000    1.000    1.00
 2  21:59   0.94   1.113   1.00    91.86    100.00    0.653    0.710    0.92
 3  21:59   0.95   1.115   0.90   141.98    100.00    1.763    1.242    1.42
 4  21:26   0.94   1.086   1.00    73.99    100.00    0.466    0.630    0.74
 5  21:26   0.94   1.086   0.92   176.89    100.00    3.730    2.109    1.77
 6  26:34   0.95   1.351   1.01    92.38    100.00    1.028    1.113    0.92
 7  26:36   0.95   1.355   0.74   144.10    100.00    1.677    1.164    1.44
 8  26:45   0.95   1.365   1.00    97.67    100.00    0.979    1.003    0.98
 9  26:43   0.95   1.363   0.74   136.88    100.00    1.670    1.220    1.37
10  30:56   0.96   1.577   1.02    55.50    100.00    0.096    0.172    0.55
11  30:56   0.96   1.579   0.63   115.22    100.00    1.412    1.226    1.15
12  35:01   0.96   1.786   1.01   116.00    100.00    0.914    0.788    1.16
13  35:01   0.96   1.788   0.56   135.84    100.00    1.749    1.287    1.36
14  34:54   0.96   1.792   1.01   129.09    100.00    0.872    0.676    1.29
15  34:54   0.97   1.795   0.56   133.48    100.00    1.808    1.355    1.33
```

TABLE 1B EXAMPLE OF SEMIVOLATILES QUANTITATION REPORT

16	38:22	0.96	1.957	1.01	120.36	100.00	0.841	0.699	1.20
17	38:22	0.96	1.957	0.51	119.50	100.00	1.624	1.359	1.19
18	38:29	0.96	1.962	1.01	122.84	100.00	0.984	0.801	1.23
19	38:29	0.96	1.965	0.51	129.90	100.00	1.459	1.123	1.30
20	39:34	0.95	2.012	1.01	142.43	100.00	0.832	0.584	1.42
21	39:34	0.95	2.015	0.50	125.26	100.00	1.593	1.272	1.25
22	46:43	0.93	2.326	1.00	143.63	100.00	0.565	0.393	1.44
23	46:43	0.93	2.334	0.43	127.06	100.00	1.961	1.543	1.27
24	49:56	0.85	2.269	1.00	129.63	100.00	0.748	0.577	1.30
25	50:21	0.84	2.283	1.00	195.06	100.00	0.183	0.094	1.95
26	20:10	0.94	1.022	1.00	101.47	100.00	1.234	1.216	1.01
27	12:39	1.50	1.025	0.98	152.35	100.00	2.244	1.473	1.52
28	12:08	0.90	0.606	0.97	80.98	100.00	0.116	0.143	0.81
29	10:53	1.02	0.607	1.67	143.83	100.00	6.374	4.431	1.44
30	15:46	0.95	0.808	1.00	125.25	100.00	0.791	0.631	1.25
31	13:39	0.91	0.684	0.98	98.66	100.00	0.173	0.176	0.99
32	12:17	1.03	0.695	1.46	129.49	100.00	1.218	0.941	1.29
33	35:37	0.96	1.827	1.01	117.97	100.00	0.773	0.655	1.18
34	34:00	1.01	1.829	0.55	138.84	100.00	1.602	1.154	1.39
35	23:35	1.02	1.285	1.01	138.60	100.00	0.343	0.248	1.39
36	31:50	1.01	1.724	1.01	161.25	100.00	0.820	0.509	1.61
37	12:16	0.90	0.600	0.99	30.75	100.00	0.279	0.907	0.31
38	11:09	1.02	0.620	1.66	218.97	100.00	0.484	0.221	2.19
39	16:30	0.93	0.836	0.99	51.08	100.00	0.433	0.849	0.51
40	16:41	0.94	0.855	1.20	210.16	100.00	0.380	0.181	2.10
41	23:22	0.95	1.190	1.01	89.42	100.00	0.653	0.730	0.89
42	24:06	0.94	1.212	0.84	142.75	100.00	0.364	0.255	1.43
43	30:22	0.91	1.497	1.00	92.70	100.00	0.568	0.613	0.93
44	27:39	1.02	1.503	0.68	153.68	100.00	0.507	0.330	1.54
45	33:35	0.96	1.723	1.01	97.95	100.00	0.558	0.570	0.98
46	32:16	1.02	1.747	0.58	135.64	100.00	0.428	0.315	1.36
47	40:15	0.95	2.044	1.01	107.94	100.00	0.534	0.495	1.08
48	38:14	1.02	2.083	0.49	167.27	100.00	0.640	0.383	1.67
49	20:07	0.94	1.020	1.00	89.81	100.00	0.718	0.800	0.90
50	20:10	0.94	1.019	0.98	138.56	100.00	1.194	0.862	1.39
51	23:44	0.95	1.206	1.01	86.24	100.00	0.354	0.410	0.86
52	22:07	1.02	1.210	0.83	147.66	100.00	1.737	1.176	1.48
53	12:57	0.90	0.643	0.98	68.84	100.00	0.660	0.959	0.69
54	12:57	0.92	0.649	1.57	117.57	100.00	1.361	1.157	1.18
55	22:31	0.94	1.141	1.00	89.71	100.00	1.021	1.139	0.90
56	22:31	0.95	1.141	0.88	136.00	100.00	1.503	1.105	1.36
57	26:12	0.95	1.327	1.01	180.49	200.00	0.966	1.070	0.90
58	26:12	0.95	1.335	0.75	150.12	100.00	1.586	1.057	1.50
59	28:54	0.96	1.478	1.01	99.93	100.00	1.220	1.221	1.00
60	28:54	0.96	1.478	0.68	136.68	100.00	1.538	1.125	1.37
61	12:37	0.90	0.629	0.97	41.34	100.00	0.129	0.312	0.41
62	12:37	0.90	0.629	1.60	202.81	100.00	3.502	1.727	2.03
63	12:45	0.90	0.637	0.97	47.51	100.00	0.158	0.333	0.48
64	12:45	0.91	0.639	1.57	183.68	100.00	3.300	1.797	1.84
65	13:17	0.91	0.664	0.98	59.04	100.00	0.187	0.317	0.59
66	13:17	0.87	0.666	1.44	151.97	100.00	2.539	1.671	1.52
67	35:00	0.96	1.792	1.01	106.41	100.00	0.265	0.249	1.06
68	35:00	0.96	1.795	0.56	96.51	100.00	1.101	1.141	0.97
69	23:43	0.95	1.206	1.01	57.28	100.00	0.440	0.768	0.57
70	23:43	0.95	1.207	0.83	182.59	100.00	2.101	1.151	1.83
71	21:27	0.95	1.090	1.00	47.17	100.00	0.451	0.955	0.47
72	21:27	0.95	1.092	0.92	172.88	100.00	1.810	1.047	1.73
73	22:47	0.95	1.159	1.01	94.98	100.00	0.257	0.271	0.95
74	22:47	0.95	1.160	0.86	144.65	100.00	1.599	1.106	1.45
75	21:34	0.95	1.095	1.00	96.55	100.00	0.202	0.209	0.97
76	21:34	0.95	1.098	0.91	130.18	100.00	1.496	1.149	1.30

TABLE 13 (CONTINUED) SEMIVOLATILES ASSOCIATED QUANTITATION REPORT

```
QUANTITATION REPORT    FILE: OPR808

DATA: OPR808.TI
08/25/86 17:52:00
SAMPLE: S3,S,OPR,00100,00,C,1000:1,08/08/86-D*
CONDS.: 1625B,

AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)
RESP. FAC. FROM AVERAGE OF WHOLE .RL

 NO  NAME
  1  164 2,2'-DIFLUOROBIPHENYL
  2  269 (D4) DI-N-OCTYLPHTHALATE
  3  369 DI-N-OCTYLPHTHALATE
  4  607 (D10) DIPHENYLAMINE
  5  707 DIPHENYLAMINE
  6  608 (D10)DIPHENYLETHER
  7  708 DIPHENYLETHER
  8  237 (D10)DIPHENYLHYDRAZINE
  9  337 1,2 DIPHENYLHYDRAZINE
 10  239 (D10)FLUORANTHENE
 11  339 FLUORANTHENE
 12  284 (D10)PYRENE
 13  384 PYRENE
 14  280 (D10)FLUORENE
 15  380 FLUORENE
 16  209 (13C6)HEXACHLOROBENZENE
 17  309 HEXACHLOROBENZENE
 18  252 (13C4)HEXACHLOROBUTADIENE
 19  352 HEXACHLOROBUTADIENE
 20  212 (13C1)HEXACHLOROETHANE
 21  312 HEXACHLOROETHANE
 22  253 (13C4)HEXACHLOROCYCLOPENTADIENE
 23  053 HEXACHLOROCYCLOPENTADIENE
 24  254 (D8)ISOPHORONE
 25  354 ISOPHORONE
 26  255 (D8)NAPHTHALENE
 27  355 NAPHTHALENE
 28  602 (D7)B-NAPHTHYLAMINE
 29  702 B-NAPHTHYLAMINE
 30  256 (D5)NITROBENZENE
 31  356 NITROBENZENE
 32  061 N-NITROSODIMETHYLAMINE
 33  063 N-NITROSODI-N-PROPYLAMINE
 34  262 (D6)N-NITROSODIPHENYLAMINE
 35  362 N-NITROSODIPHENYLAMINE
 36  265 (D5)PHENOL
 37  365 PHENOL
 38  603 (D7) A-PICOLINE
 39  703 A-PICOLINE
 40  610 (D5)STYRENE
 41  710 STYRENE
 42  609 (D3)A-TERPINEOL
 43  509 A-TERPINEOL
 44  208 (D3)1,2,4 TRICHLOROBENZENE
 45  308 1,2,4 TRICHLOROBENZENE
 46  222 (D2) 4-CHLORO-3-METHYLPHENOL
 47  322 4-CHLORO-3-METHYLPHENOL
 48  224 (D4) 2-CHLOROPHENOL
 49  324 2-CHLOROPHENOL
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TABLE 1B (CONTINUED)   SEMIVOLATILES ASSOCIATED QUANTITATION REPORT

```
50   231 (D3) 2,4 DICHLOROPHENOL
51   331 2,4 DICHLOROPHENOL
52   259 (D3)2,4 DINITROPHENOL
53   359 2,4 DINITROPHENOL
54   260 (D2) 2-METHYL-4,6-DINITROPHENOL
55   360 2-METHYL-4,6-DINITROPHENOL
56   257 (D4) 2-NITROPHENOL
57   357 2-NITROPHENOL
58   258 (D4) 4-NITROPHENOL
59   358 4-NITROPHENOL
60   264 (13C6)PENTACHLOROPHENOL
61   364 PENTACHLOROPHENOL
62   221 (D2)2,4,6 TRICHLOROPHENOL
63   321 2,4,6 TRICHLOROPHENOL
64   234 (D2) 2,4 DIMETHYLPHENOL
65   334 2,4 DIMETHYLPHENOL
66   531 2,4,5 TRICHLOROPHENOL
67   530 2,3,6 TRICHLOROPHENOL
68   529 1,2,3 TRICHLOROBENZENE
69   518 N-TETRADECANE
70   520 N-OCTADECANE
71   522 N-DOCOSANE
72   524 N-HEXACOSANE
73   525 N-OCTACOSANE
74   528 CARBAZOLE

NO   M/E   SCAN   TIME   REF   RRT    METH      AREA(HGHT)  AMOUNT             %TOT
 1   190   1060   18:33   1   1.000   A BB      107460.    100.000 UG/ML     1.19
 2   153   2067   36:10   1   1.950   A BB      143775.    135.457 UG/ML     1.61
 3   149   2068   36:11   2   1.000   A BB      240328.    135.396 UG/ML     1.61
 4   179   1309   22:54   1   1.235   QEDT       64139.     85.889 UG/ML     1.02
 5   169   1314   23:00   4   1.004   RXRY      120649.    137.544 UG/ML     1.63
 6   180   1105   19:20   1   1.042   A?BB       55859.     84.978 UG/ML     1.01
 7   170   1110   19:25   6   1.005   QEDT       85654.    138.999 UG/ML     1.65
 8    82   1310   22:55   1   1.236   A BB       98702.     82.535 UG/ML     0.98
 9    77   1316   23:02   8   1.005   A BB      172069.    157.483 UG/ML     1.87
10   212   1656   28:59   1   1.562   A BB      109709.     98.307 UG/ML     1.17
11   202   1660   29:03  10   1.002   A BB      179722.    147.826 UG/ML     1.75
12   212   1694   29:39   1   1.598   A BB      109394.    100.739 UG/ML     1.20
13   202   1698   29:43  12   1.002   A BB      185183.    141.871 UG/ML     1.68
14   176   1274   22:18   1   1.202   QEDT       80181.    101.325 UG/ML     1.20
15   166   1280   22:24  14   1.005   A BB      140832.    129.170 UG/ML     1.53
16   292   1391   24:21   1   1.312   A?BB       18979.     94.277 UG/ML     1.12
17   284   1392   24:22  16   1.001   RXRY       34019.    123.320 UG/ML     1.46
18   231    915   16:01   1   0.863   A BB        9961.     72.803 UG/ML     0.86
19   225    916   16:02  18   1.001   RXRY       28306.    150.926 UG/ML     1.79
20   204    740   12:57   1   0.698   A BB        3245.     35.116 UG/ML     0.42
21   201    740   12:57  20   1.000   RXRY       13715.    222.595 UG/ML     2.64
22   241   1041   18:13   1   0.982   A BB        1697.     55.062 UG/ML     0.65
23   237   1041   18:13   1   0.982   A BB        8490.     67.086 UG/ML     0.80
24    88    802   14:02   1   0.757   QEDT       95203.     87.342 UG/ML     1.04
25    82    809   14:09  24   1.009   RXRY      160471.    142.082 UG/ML     1.69
26   136    874   15:18   1   0.825   A BB      105146.     79.885 UG/ML     0.95
27   128    877   15:21  26   1.003   RXRY      180320.    143.467 UG/ML     1.70
28   150   1248   21:50   1   1.177   A?BB       14520.     46.703 UG/ML     0.55
29   143   1251   21:54  28   1.002   A BB       49135.    316.879 UG/ML     3.76
30   128    758   13:16   1   0.715   A?BB       23684.     83.687 UG/ML     0.99
31   123    761   13:19  30   1.004   A BB       35461.    143.975 UG/ML     1.71
32    74    234    4:06   1   0.221   A BB       41740.    107.944 UG/ML     1.28
33    70    748   13:05   1   0.706   QEDT       63489.    130.887 UG/ML     1.55
34   175   1312   22:58   1   1.238   A?BB       7[illegible]34.     85.454 UG/ML     1.01
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        TABLE 1B (CONTINUED)    SEMIVOLATILES ASSOCIATED QUANTITATION REPORT
35  169  1314  23:00  34  1.002  RXRY     103729.    132.720 UG/ML  1.57
36   71   634  11:06   1  0.598  QEDT      13868.     87.694 UG/ML  1.04
37   94   636  11:08  36  1.003  RXRY      71975.    127.512 UG/ML  1.51
38  100   339   5:56   1  0.320  A BB      38307.     69.071 UG/ML  0.82
39   93   344   6:01  38  1.015  A BB      41582.    136.715 UG/ML  1.62
40  109   473   8:17   1  0.446  A BB      25038.     35.453 UG/ML  0.42
41  104   476   8:20  40  1.006  A BB      54828.    201.822 UG/ML  2.39
42  NOT FOUND
43   59   891  15:36   1  0.841  A?BB      79586.    131.130 UG/ML  1.56
44  183   868  15:11   1  0.819  QEDT      39227.     81.887 UG/ML  0.97
45  180   870  15:13  44  1.002  RXRY      63032.    143.393 UG/ML  1.70
46  109   999  17:29   1  0.942  A BB      36887.     96.536 UG/ML  1.15
47  107   998  17:28  46  0.999  RXRY      57225.    136.043 UG/ML  1.61
48  132   631  11:03   1  0.595  A BB      42815.     83.808 UG/ML  0.99
49  128   634  11:06  48  1.005  RXRY      61913.    136.137 UG/ML  1.62
50  167   862  15:05   1  0.813  QEDT      19970.     81.558 UG/ML  0.97
51  162   863  15:06  50  1.001  QEDT      42143.    129.050 UG/ML  1.53
52  187  1212  21:13   1  1.143  QEDT       2300.     21.528 UG/ML  0.26
53  184  1216  21:17  52  1.003  RXRY       2709.    133.703 UG/ML  1.59
54  200  1309  22:54   1  1.235  QEDT      12529.     77.720 UG/ML  0.92
55  198  1310  22:55  54  1.001  QEDT      17624.    135.614 UG/ML  1.61
56  143   814  14:15   1  0.768  A BB      24818.     87.204 UG/ML  1.03
57  139   816  14:17  56  1.002  QEDT      34296.    133.171 UG/ML  1.58
58  143  1246  21:48   1  1.175  A?BV       9620.     69.945 UG/ML  0.83
59  139  1246  21:48  58  1.000  RXRY      10847.    108.875 UG/ML  1.29
60  272  1429  25:00   1  1.348  A BB       7731.     64.074 UG/ML  0.76
61  266  1430  25:01  60  1.001  RXRY      10703.    143.443 UG/ML  1.70
62  200  1061  18:34   1  1.001  A?BV      33775.    105.940 UG/ML  1.26
63  196  1062  18:35  62  1.001  RXRY      61945.    143.180 UG/ML  1.70
64  124   842  14:44   1  0.794  QEDT      17300.     44.540 UG/ML  0.53
65  122   843  14:45  64  1.001  RXRY      26104.    131.328 UG/ML  1.56
66  196  1069  18:42   1  1.008  QEDT      40489.    142.543 UG/ML  1.69
67  196  1092  19:07   1  1.030  QEDT      40067.    148.899 UG/ML  1.77
68  180   910  15:55   1  0.858  A BB      55181.    116.012 UG/ML  1.38
69   55  1106  19:21   1  1.043  A BB      23518.    109.258 UG/ML  1.30
70   55  1455  25:28   1  1.373  A BB      29798.    136.221 UG/ML  1.62
71   55  1743  30:30   1  1.644  A BB      28663.    143.459 UG/ML  1.70
72   55  1988  34:47   1  1.875  QEDT      30552.    161.644 UG/ML  1.92
73   55  2099  36:44   1  1.980  QEDT      29190.    151.641 UG/ML  1.80
74 .167  1492  26:07   1  1.408  A?BB     140684.    140.405 UG/ML  1.67

NO  RET(L) RATIO RRT(L) RATIO     AMNT   AMNT(L)   R.FAC R.FAC(L) RATIO
 1  18:52  0.98  1.000  1.00    100.00   100.00   1.000   1.000   1.00
 2  37:33  0.96  1.924  1.01    135.46   100.00   1.338   0.988   1.35
 3  29:17  1.24  1.927  0.52    135.40   100.00   1.672   1.235   1.35
 4  19:07  1.20  1.222  1.01     85.89   100.00   0.597   0.695   0.86
 5  25:27  0.90  1.227  0.82    137.54   100.00   1.881   1.368   1.38
 6  15:37  1.24  1.041  1.00     84.98   100.00   0.520   0.612   0.85
 7  15:34  1.25  1.046  0.96    139.00   100.00   1.533   1.103   1.39
 8  24:11  0.95  1.225  1.01     82.53   100.00   0.918   1.113   0.83
 9  24:17  0.95  1.229  0.82    157.48   100.00   1.743   1.107   1.57
10  24:11  1.20  1.546  1.01     98.31   100.00   1.021   1.039   0.98
11  23:30  1.24  1.550  0.65    147.83   100.00   1.638   1.108   1.48
12  24:11  1.23  1.580  1.01    100.74   100.00   1.018   1.011   1.01
13  24:03  1.24  1.585  0.63    141.87   100.00   1.693   1.193   1.42
14  18:06  1.23  1.194  1.01    101.33   100.00   0.746   0.736   1.01
15  18:00  1.24  1.199  0.84    129.17   100.00   1.756   1.360   1.29
16  20:23  1.19  1.305  1.01     94.28   100.00   0.177   1.187   0.94
17  19:37  1.24  1.305  0.77    123.32   100.00   1.792    .453   1.23
18  13:11  1.22  0.872  0.99     72.80   100.00   0.093   0.127   0.73
19  12:42  1.26  0.872  1.15    150.93   100.00   2.842   1.883   1.51
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TABLE 1B (CONTINUED) SEMIVOLATILES ASSOCIATED QUANTITATION REPORT

20	10:36	1.22	0.706	0.99	35.12	100.00	0.030	0.086	0.35
21	10:14	1.26	0.706	1.42	222.59	100.00	4.226	1.899	2.23
22	15:06	1.21	0.986	1.00	55.06	100.00	0.016	0.029	0.55
23	12:01	1.52	0.986	1.00	67.09	100.00	0.079	0.118	0.67
24	8:29	1.65	0.761	0.99	87.34	100.00	0.886	1.014	0.87
25	11:14	1.26	0.768	1.31	142.08	100.00	1.686	1.186	1.42
26	12:18	1.24	0.830	0.99	79.89	100.00	0.978	1.225	0.80
27	12:11	1.26	0.833	1.20	143.47	100.00	1.715	1.195	1.43
28	17:40	1.24	1.168	1.01	46.70	100.00	0.135	0.289	0.47
29	17:39	1.24	1.171	0.86	316.88	100.00	3.384	1.068	3.17
30	10:38	1.25	0.720	0.99	83.69	100.00	0.220	0.263	0.84
31	10:32	1.26	0.722	1.39	143.97	100.00	1.497	1.040	1.44
32	2:58	1.38	0.267	0.83	107.94	100.00	0.388	0.360	1.08
33	10:50	1.21	0.710	0.99	130.89	100.00	0.591	0.451	1.31
34	18:36	1.23	1.228	1.01	85.45	100.00	0.656	0.768	0.85
35	18:34	1.24	1.230	0.81	132.72	100.00	1.471	1.108	1.33
36	8:46	1.27	0.614	0.97	87.69	100.00	0.129	0.147	0.88
37	8:41	1.28	0.617	1.63	127.51	100.00	5.190	4.070	1.28
38	2:13	2.67	0.360	0.89	69.07	100.00	0.356	0.516	0.69
39	2:36	2.31	0.376	2.70	136.72	100.00	1.085	0.794	1.37
40	10:16	0.81	0.478	0.93	35.45	100.00	0.233	0.657	0.35
41	10:21	0.81	0.481	2.09	201.82	100.00	2.190	1.085	2.02
42	17:36		0.844			100.00		0.462	
43	12:22	1.26	0.843	1.00	131.13	100.00	0.741	0.565	1.31
44	17:17	0.88	0.830	0.99	81.89	100.00	0.365	0.446	0.82
45	12:04	1.26	0.826	1.21	143.39	100.00	1.607	1.121	1.43
46	11:03	1.58	0.938	1.00	96.54	100.00	0.343	0.356	0.97
47	13:51	1.26	0.938	1.07	136.04	100.00	1.551	1.140	1.36
48	8:50	1.25	0.612	0.97	83.81	100.00	0.398	0.475	0.84
49	8:45	1.27	0.615	1.63	136.14	100.00	1.446	1.062	1.36
50	12:01	1.25	0.814	1.00	81.56	100.00	0.186	0.228	0.82
51	11:55	1.27	0.816	1.23	129.05	100.00	2.110	1.635	1.29
52	14:24	1.47	1.131	1.01	21.53	100.00	0.021	0.099	0.22
53	17:04	1.25	1.130	0.89	133.70	100.00	1.178	0.881	1.34
54	15:36	1.47	1.224	1.01	77.72	100.00	0.117	0.150	0.78
55	18:32	1.24	1.225	0.82	135.61	100.00	1.407	1.037	1.36
56	11:26	1.25	0.772	0.99	87.20	100.00	0.231	0.265	0.87
57	13:58	1.02	0.774	1.29	133.17	100.00	1.382	1.038	1.33
58	23:11	0.94	1.159	1.01	69.95	100.00	0.090	0.128	0.70
59	22:44	0.96	1.162	0.86	108.88	100.00	1.128	1.036	1.09
60	17:21	1.44	1.338	1.01	64.07	100.00	0.072	0.112	0.64
61	17:23	1.44	1.341	0.75	143.44	100.00	1.384	0.965	1.43
62	14:56	1.24	0.999	1.00	105.94	100.00	0.314	0.297	1.06
63	14:49	1.25	1.000	1.00	143.18	100.00	1.834	1.281	1.43
64	16:18	0.90	0.799	0.99	44.54	100.00	0.161	0.361	0.45
65	11:36	1.27	0.800	1.25	131.33	100.00	1.509	1.149	1.31
66	18:17	1.02	1.005	1.00	142.54	100.00	0.377	0.264	1.43
67	18:42	1.02	1.027	1.00	148.90	100.00	0.373	0.250	1.49
68	15:36	1.02	0.863	0.99	116.01	100.00	0.514	0.443	1.16
69	18:56	1.02	1.039	1.00	109.26	100.00	0.219	0.200	1.09
70	24:59	1.02	1.364	1.01	136.22	100.00	0.277	0.204	1.36
71	30:00	1.02	1.630	1.01	143.46	100.00	0.267	0.186	1.43
72	34:33	1.01	1.855	1.01	161.64	100.00	0.284	0.176	1.62
73	38:09	0.96	1.958	1.01	151.64	100.00	0.272	0.179	1.52
74	25:40	1.02	1.398	1.01	140.41	100.00	1.309	0.932	1.40

TABLE 1c EXAMPLE OF DIOXIN QUANTITATION REPORT

```
QUANTITATION REPORT    FILE: TCDD074

DATA:  TCDD074
02/06/84 21:35:00
SAMPLE: 02,S,CAL,01000,00,D,NA:NA,NAS
CONDS.: 613E,60M,0.25MM,3*100,100-250*15,15*250,30CM/SS
FORMULA:                    INSTRUMENT: 02                   WEIGHT:     0.000
SUBMITTED BY:               ANALYST: SH                      ACCT.NO.:
  AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)

 NO  NAME
  1  184   2,3,7,8-TCDD-37CLA
  2  429   2,3,7,8-TCDD-13C1[illegible]
  3  829   2,3,7,8-TCDD

 NO   M/E   SCAN    TIME   REF  [illegible]ETH      AREA(HGHT)  AMOUNT            %TOT
  1   328   1517   24:24    1   1.[illegible] BB        15020.   1000.000 NG/ML 33.37
  2   332   1515   24:22    1   0.9[illegible] A BB     13146.    985.413 NG/ML 32.88
  3   320   1517   24:24    2   1.001  A BB        11382.   1011.624 NG/ML 33.75

 NO  RET(L) RATIO RRT(L) RATIO       AMNT      AMNT(L)    R.FAC R.FAC(L) RATIO
 01  24:26  1.00  1.000  1.00    1000.00     1000.00    1.000   1.000    1.00
 02  24:24  1.00  0.999  1.00     985.41     1000.00    0.875   0.888    0.99
 03  24:26  1.00  1.001  1.00    1011.62     1000.00    0.758   0.749    1.01
```

TABLE 2 - Quantitation Report Magnetic Tape Transmittal Form Description

The main purpose of the tape transmittal form is to ensure the complete and correct data processing of a tape volume (reel). Depending upon the number of files per volume at least one tape transmittal form must accompany each tape volume sent to the Sample Control Center. Field descriptions are as follows.

Laboratory	:	The laboratory name.
Return Tape To	:	The address to which the tape volume is to be returned after processing by the SCC.
External Tape #	:	The tape number assigned by the SCC.
Lab Tape ID	:	The laboratory tape identification.
Tape Density	:	Either 800 or 1600 bpi.
Block Size	:	800 decimal words or bytes per block.
Number of Files	:	The number of Quantitation report files on the tape.
Contact Person and Phone Number	:	Whom to contact at the laboratory, regarding any difficulties in the processing of the tape volume, and their phone number including area code.
File Position	:	The relative file position of the Quantitation Report File on the tape volume. The first file on the tape has a relative file position of 1. The second file on the tape has a relative file position of 2.
Lab Data File	:	The laboratory data file identification.

Instrument ID : The laboratory assigned instrument specific 2 digit alphanumeric code that is used to identify a GC/MS instrument.

Note: All quantitation files for calibration, precision and recovery, standards and blanks will be tracked by this instrument number within a given laboratory. Changing of this instrument number by the laboratory would necessitate the submittal of new calibration and initial precision and recovery quantitation reports by the laboratory.

EPA Sample Number : The 5 digit sample number for the sample:

Sample Type	Sample Number
IPR 20	00020
IPR 100	00100
BLK	00000
BLK 1000	01000
CAL 10	00010
CAL 20	00020
CAL 50	00050
CAL 100	00100
CAL 200	00200
CAL 1000	01000
CAL 5000	05000
CAL 20000	20000
CAL 40000	40000
EPA	SCC Assigned Sample Number
FBK	SCC Assigned Sample Number
VER	00100
VER 1000	01000

Bottle Number : The 2 digit (numeric) bottle number used to identify each individual aliquot of the sample.

Sample Type	Bottle Number
EPA	01-98
EPA (analysis of TCLP Leachate)	99
All Others	00

Type : The 3 position EGLD code signifying the sample or quantitation type. Required.

Sample Type	Meaning
IPR	Initial Precision and Accuracy
BLK	Blank
CAL	Calibration
EPA	EPA Sample
FBK	Field Blank
OPR	Ongoing Precision and Accuracy
VER	Calibration Verification for extraction methods

Fraction : 1 position code:

A = Acid
B = Base/Neutral
C = Combined Acid Base/Neutral
D = Dioxin
V = Volatile
P = Pesticide

N/A if not applicable.

Conc/Dilu : The concentration or dilution ratio of the sample fraction before analysis. N/A if not applicable.

Date Analyzed : The date of analysis.

USEPA Industrial Technology Division Revision: C

Quantitation Report Magnetic Tape Transmittal Form Date: 10 October 86

Laboratory: ______________________ External Tape #: ______________

Return Tape To: ______________________ Lab Tape ID: ______________

______________________ Tape Density (BPI): ______________

______________________ Block Size: ______________

______________________ Number of Files: ______________

Contact Person and Phone Number: ______________________ (___) _____ - _____

File Position	Lab Data File	Instrument ID	EPA Sample #	Bottle #	Type	Fraction	Conc Dilu	Date Analyzed

The data recorded on this tape have been verified and are true and complete.

Date: ______________ Analyst: ______________________ QA: ______________________

Table 2a - Quantitation Report Magnetic Tape Additional Files Transmittal Form

This form is only to be used when there are more than 14 quantitation report files on a tape and it must be used with a Quantitation Report Magnetic Tape Transmittal Form. As many Additional Files Transmittal Forms can be used to accommodate all of the files on the tape. The field definitions are identical to those on the Quantitation Report Magnetic Tape Transmittal Form.

Quantitation Report Magnetic Tape
Additonal Files Transmittal Form

(This form must be used with the Quantitation Report Magnetic Tape Transmittal Form)

Laboratory: ______________________ External Tape #: ______________

File Position	Lab Data File	Instrument ID	EPA Sample #	Bottle #	Type	Fraction	Conc Dilu	Date Analyzed

TAB 3

Report Form I

Lab ____________________ Sample Number ____________________

File Position ____________________ Sample Type ____________________

EGD Number				Retention Time		Rel Ret Time		Peak	Amount (ug/mL)		Response Factor		
Data	Ref	m/z	Scan	Data	Library	Data	Library	area	Data	Library	Data	Library	Note

Comments which reference note above

COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE	CODE DESCRIPTION				
04	001	B	ACENAPHTHENE	P	0154	Y
04	002	V	ACROLEIN	P	0056	Y
04	003	V	ACRYLONITRILE	P	0053	Y
04	004	V	BENZENE	P	0078	Y
04	005	B	BENZIDINE	P	0184	Y
04	006	V	CARBON TETRACHLORIDE	P	0047	Y
04	007	V	CHLOROBENZENE	P	0112	Y
04	008	B	1,2,4-TRICHLOROBENZENE	P	0180	Y
04	009	B	HEXACHLOROBENZENE	P	0284	Y
04	010	V	1,2-DICHLOROETHANE	P	0062	Y
04	011	V	1,1,1-TRICHLOROETHANE	P	0097	Y
04	012	B	HEXACHLOROETHANE	P	0201	Y
04	013	V	1,1-DICHLOROETHANE	P	0063	Y
04	014	V	1,1,2-TRICHLOROETHANE	P	0083	Y
04	015	V	1,1,2,2-TETRACHLOROETHANE	P	0083	Y
04	016	V	CHLOROETHANE	P	0064	Y
04	017	V	BIS (CHLOROMETHYL) ETHER (NR)	P		Y
04	018	B	BIS(2-CHLOROETHYL)ETHER	P	0093	Y
04	019	V	2-CHLOROETHYLVINYL ETHER	P	0106	Y
04	020	B	2-CHLORONAPHTHALENE	P	0162	Y
04	021	A	2,4,6-TRICHLOROPHENOL	P	0196	Y
04	022	A	4-CHLORO-3-METHYLPHENOL	P	0107	Y
04	023	V	CHLOROFORM	P	0085	Y
04	024	A	2-CHLOROPHENOL	P	0128	Y
04	025	B	1,2-DICHLOROBENZENE	P	0146	Y
04	026	B	1,3-DICHLOROBENZENE	P	0146	Y
04	027	B	1,4-DICHLOROBENZENE	P	0146	Y
04	028	B	3,3'-DICHLOROBENZIDINE	P	0252	Y
04	029	V	1,1-DICHLOROETHENE	P	0061	Y
04	030	V	TRANS-1,2-DICHLOROETHENE	P	0061	Y
04	031	A	2,4-DICHLOROPHENOL	P	0162	Y
04	032	V	1,2-DICHLOROPROPANE	P	0063	Y
04	033	V	T-1,3-DICHLOROPROPENE	P	0075	Y
04	034	B	2,4-DIMETHYLPHENOL	P	0122	Y
04	035	B	2,4-DINITROTOLUENE	P	0165	Y
04	036	B	2,6-DINITROTOLUENE	P	0165	Y
04	037	B	1,2-DIPHENYLHYDRAZINE	P	0077	Y
04	038	V	ETHYLBENZENE	P	0106	Y
04	039	B	FLUORANTHENE	P	0202	Y
04	040	B	4-CHLOROPHENYL PHENYL ETHER	P	0204	Y
04	041	B	4-BROMOPHENYL PHENYL ETHER	P	0248	Y
04	042	B	BIS (2-CHLOROISOPROPYL) ETHER	P	0121	Y
04	043	B	BIS (2-CHLOROETHOXY) METHANE	P	0093	Y
04	044	V	METHYLENE CHLORIDE	P	0084	Y
04	045	V	CHLOROMETHANE	P	0050	Y
04	046	V	BROMOMETHANE	P	0096	Y
04	047	V	BROMOFORM	P	0173	Y
04	048	V	BROMODICHLOROMETHANE	P	0083	Y
04	049	V	TRICHLOROFLUOROMETHANE (NR)	P		Y
04	050	V	DICHLORODIFLUOROMETHANE (NR)	P		Y

COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	051	V	DIBROMOCHLOROMETHANE	P	0129	Y
04	052	B	HEXACHLORO-1,3-BUTADIENE	P	0225	Y
04	053	B	HEXACHLOROCYCLOPENTADIENE	P	0237	Y
04	054	B	ISOPHORONE	P	0082	Y
04	055	B	NAPHTHALENE	P	0128	Y
04	056	B	NITROBENZENE	P	0123	Y
04	057	A	2-NITROPHENOL	P	0139	Y
04	058	A	4-NITROPHENOL	P	0139	Y
04	059	A	2,4-DINITROPHENOL	P	0184	Y
04	060	A	2-METHYL-4,6-DINITROPHENOL	P	0198	Y
04	061	B	N-NITROSODIMETHYLAMINE	P	0074	Y
04	062	B	N-NITROSODIPHENYLAMINE	P	0169	Y
04	063	B	N-NITROSODI-N-PROPYLAMINE	P	0070	Y
04	064	A	PENTACHLOROPHENOL	P	0266	Y
04	065	B	PHENOL	P	0094	Y
04	066	B	BIS (2-ETHYLHEXYL) PHTHALATE	P	0149	Y
04	067	B	BUTYL BENZYL PHTHALATE	P	0149	Y
04	068	B	DI-N-BUTYL PHTHALATE	P	0149	Y
04	069	B	DI-N-OCTYL PHTHALATE	P	0149	Y
04	070	B	DIETHYL PHTHALATE	P	0149	Y
04	071	B	DIMETHYL PHTHALATE	P	0163	Y
04	072	B	BENZO(A)ANTHRACENE	P	0228	Y
04	073	B	BENZO(A)PYRENE	P	0252	Y
04	074	B	BENZO(B)FLUORANTHENE	P	0252	Y
04	075	B	BENZO(K)FLUORANTHENE	P	0252	Y
04	076	B	CHRYSENE	P	0228	Y
04	077	B	ACENAPHTHYLENE	P	0152	Y
04	078	B	ANTHRACENE	P	0178	Y
04	079	B	BENZO(GHI)PERYLENE	P	0276	Y
04	080	B	FLUORENE	P	0166	Y
04	081	B	PHENANTHRENE	P	0178	Y
04	082	B	DIBENZO(A,H)ANTHRACENE	P	0278	Y
04	083	B	INDENO(1,2,3-CD)PYRENE	P	0276	Y
04	084	B	PYRENE	P	0202	Y
04	085	V	TETRACHLOROETHENE	P	0164	Y
04	086	V	TOLUENE	P	0092	Y
04	087	V	TRICHLOROETHENE	P	0095	Y
04	088	V	VINYL CHLORIDE	P	0062	Y
04	089	P	ALDRIN	P		Y
04	090	P	DIELDRIN	P		Y
04	091	P	CHLORDANE	P		Y
04	092	P	4,4'-DDT	P		Y
04	093	P	4,4'-DDE	P		Y
04	094	P	4,4'-DDD	P		Y
04	095	P	ALPHA-ENDOSULFAN	P		Y
04	096	P	BETA-ENDOSULFAN	P		Y
04	097	P	ENDOSULFAN SULFATE	P		Y
04	098	P	ENDRIN	P		Y
04	099	P	ENDRIN ALDEHYDE	P		Y
04	100	P	HEPTACHLOR	P		Y

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COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	101	P	HEPTACHLOR EPOXIDE	P		Y
04	102	P	ALPHA-BHC	P		Y
04	103	P	BETA-BHC	P		Y
04	104	P	GAMMA-BHC	P		Y
04	105	P	DELTA-BHC	P		Y
04	106	P	PCB-1242	P		Y
04	107	P	PCB-1254	P		Y
04	108	P	PCB-1221	P		Y
04	109	P	PCB-1232	P		Y
04	110	P	PCB-1248	P		Y
04	111	P	PCB-1260	P		Y
04	112	P	PCB-1016	P		Y
04	113	P	TOXAPHENE	P		Y
04	129	D	2,3,7,8-TCDD	P	0320	Y
04	130	V	XYLENES	P		Y
04	150	A	PHENOL-D5	S		Y
04	151	A	PENTAFLUOROPHENOL	S		Y
04	152	V	PENTAFLOUROBENZENE	S		Y
04	153	A	TRIFLUORO-M-CRESOL	S		Y
04	154	V	2,2-DIFLUOROTETRACHLOROETHANE	S		Y
04	155	B	2-FLUOROBIPHENYL	S		Y
04	156	B	1-FLUORONAPHTHALENE	S		Y
04	157	A	2-FLOUROPHENOL	S		Y
04	158	B	2-FLUORONAPHTHALENE	S		Y
04	159	B	PYRIDINE-D5	S		Y
04	160	B	ANILINE-D5	S		Y
04	161	B	NAPHTHALENE-D8	S		Y
04	162	V	TOLUENE-D8	S		Y
04	163	B	NITROBENZENE-D5	S		Y
04	164	B	2,2'-DIFLUOROBIPHENYL	I	0190	Y
04	165	V	BENZENE-D6	S		Y
04	166	B	DECAFLUOROBIPHENYL	S		Y
04	167	V	M-DIFLUOROBENZENE	S		Y
04	168	V	METHYLENE CHLORIDE-D2	S		Y
04	169	V	1,1,2,2-TETRACHLOROETHANE-D2	S		Y
04	170	V	ETHYLBENZENE-D10	S		Y
04	172	V	1,2 DICHLOROETHANE-D4	S		Y
04	173	V	2,2 DICHLOPROPANE-D6	S		Y
04	174	V	CHLOROBENZENE-D5	S		Y
04	175	B	1,2 DICHLOROBENZENE-D4	S		Y
04	176	B	CHRYSENE D12	S		Y
04	177	B	FLUORENE D10	S		Y
04	178	A	2-NITROPHENOL D4	S		Y
04	179	B	DI-N-BUTYL-PHTHALATE-D4	S		Y
04	180	B	4-FLUOROANILINE	S		Y
04	181	V	BROMOCHLOROMETHANE	I	0128	Y
04	182	V	2-BROMO-1-CHLOROPROPANE	I	0077	Y
04	183	V	1,4-DICHLOROBUTANE	I	0055	Y
04	184	D	2,3,7,8-TCDD-37CL4	I	0328	Y
04	185	V	BROMOFLUOROBENZENE	I	0095	Y

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COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	186	B	DFTPP	I	0198	N
04	187	P	2'-CHLOROBIPHENYL-D5	S		N
04	188	P	3',4',5'-TRICHLOROBIPHENYL-D5	S		N
04	189	P	3,3',4,4'-TETRACHLOROBIPHEN-D6	S		N
04	201	B	ACENAPHTHENE-D10	D	0164	Y
04	202	V	ACROLEIN-D4	D	0060	Y
04	203	V	ACRYLONITRILE-D3	D	0056	Y
04	204	V	BENZENE-D6	D	0084	Y
04	205	B	BENZIDINE (RINGS-D8)	D	0192	Y
04	206	V	CARBON TETRACHLORIDE-13C	D	0048	Y
04	207	V	CHLOROBENZENE-D5	D	0117	Y
04	208	B	1,2,4-TRICHLOROBENZENE-D3	D	0183	Y
04	209	B	HEXACHLOROBENZENE-13C6	D	0292	Y
04	210	V	1,2-DICHLOROETHANE-D4	D	0067	Y
04	211	V	1,1,1-TRICHLOROETHANE-D3	D	0102	Y
04	212	B	HEXACHLOROETHANE-1-13C	D	0204	Y
04	213	V	1,1-DICHLOROETHANE-2,2,2-D3	D	0066	Y
04	214	V	1,1,2-TRICHLOROETHANE-13C2	D	0084	Y
04	215	V	1,1,2,2-TETRACHLOROETHANE-D2	D	0084	Y
04	216	V	CHLOROETHANE-D5	D	0071	Y
04	217	V		D		N
04	218	B	BIS(2-CHLOROETHYL)ETHER-D8	D	0101	Y
04	219	V		D	0113	N
04	220	B	2-CHLORONAPHTHALENE-D7	D	0169	Y
04	221	A	2,4,6-TRICHLOROPHENOL-3,5-D2	D	0200	Y
04	222	A	4-CHLORO-3-METHYLPHENOL-2,6-D2	D	0109	Y
04	223	V	CHLOROFORM-13C	D	0086	Y
04	224	A	2-CHLOROPHENOL-3,4,5,6-D4	D	0132	Y
04	225	B	1,2-DICHLOROBENZENE-D4	D	0152	Y
04	226	B	1,3-DICHLOROBENZENE-D4	D	0152	Y
04	227	B	1,4-DICHLOROBENZENE-D4	D	0152	Y
04	228	B	3,3'-DICHLOROBENZIDINE-D6	D	0258	Y
04	229	V	1,1-DICHLOROETHENE-D2	D	0065	Y
04	230	V	TRANS-1,2-DICHLOROETHENE-D2	D	0065	Y
04	231	A	2,4-DICHLOROPHENOL-3,5,6-D3	D	0167	Y
04	232	V	1,2-DICHLOROPROPANE-D6	D	0067	Y
04	233	V	T-1,3-DICHLOROPROPENE-1,2-D4	D	0079	Y
04	234	B	2,4-DIMETHYLPHENOL-3,5,6-D3	D	0125	Y
04	235	B	2,4-DINITROTOLUENE-3,5,6-D3	D	0168	Y
04	236	B	2,6-DINITROTOLUENE-A,A,A-D3	D	0167	Y
04	237	B	1,2-DIPHENYL-D10-HYDRAZINE	D	0082	Y
04	238	V	ETHYLBENZENE-D10	D	0116	Y
04	239	B	FLUORANTHENE-D10	D	0212	Y
04	240	B	4-CHLOROPHENYL PHENYL-D5 ETHER	D	0209	Y
04	241	B		D		N
04	242	B	BIS(2-CHLOROISOPROPYL)ETHERD12	D	0131	Y
04	243	B		D		N
04	244	V	METHYLENE CHLORIDE-D2	D	0088	Y
04	245	V	CHLOROMETHANE-D3	D	0053	Y
04	246	V	BROMOMETHANE-D3	D	0099	Y

COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	247	V	BROMOFORM-13C	D	0176	Y
04	248	V	BROMODICHLOROMETHANE-13C	D	0086	Y
04	249	V		D		N
04	250	V		D		N
04	251	V	DIBROMOCHLOROMETHANE-13C	D	0130	Y
04	252	B	HEXACHLORO-1,3-BUTADIENE-13C4	D	0231	Y
04	253	B	HEXACHLOROCYCLOPENTADIENE-13C4	D	0241	Y
04	254	B	ISOPHORONE-D8	D	0088	Y
04	255	B	NAPHTHALENE-D8	D	0136	Y
04	256	B	NITROBENZENE-D5	D	0128	Y
04	257	A	2-NITROPHENOL-3,4,5,6-D4	D	0143	Y
04	258	A	4-NITROPHENOL-2,3,5,6-D4	D	0143	Y
04	259	A	2,4-DINITROPHENOL-3,5,6-D3	D	0187	Y
04	260	A	2-METHYL-4,6-DINITROPHENOL-D2	D	0200	Y
04	261	B		D		N
04	262	B	N-NITROSODIPHENYLAMINE-D6	D	0175	Y
04	263	B		D		N
04	264	A	PENTACHLOROPHENOL-13C6	D	0272	Y
04	265	B	PHENOL-2,3,4,5,6-D5	D	0071	Y
04	266	B	BIS(2-ETHYLHEXYL)PHTHALATE-D4	D	0153	Y
04	267	B		D		N
04	268	B	DI-N-BUTYL PHTHALATE-D4	D	0153	Y
04	269	B	DI-N-OCTYL PHTHALATE-D4	D	0153	Y
04	270	B	DIETHYL PHTHALATE-3,4,5,6-D4	D	0153	Y
04	271	B	DIMETHYL PHTHALATE-3,4,5,6-D4	D	0167	Y
04	272	B	BENZO(A)ANTHRACENE-D12	D	0240	Y
04	273	B	BENZO(A)PYRENE-D12	D	0264	Y
04	274	B	BENZO(B)FLUORANTHENE-D12	D	0264	Y
04	275	B	BENZO(K)FLUORANTHENE-D12	D	0264	Y
04	276	B	CHRYSENE-D12	D	0240	Y
04	277	B	ACENAPHTHYLENE-D8	D	0160	Y
04	278	B	ANTHRACENE-D10	D	0188	Y
04	279	B	BENZO(GHI)PERYLENE-D12	D	0288	Y
04	280	B	FLUORENE-D10	D	0176	Y
04	281	B	PHENANTHRENE-D10	D	0188	Y
04	282	B		D		N
04	283	B		D		N
04	284	B	PYRENE-D10	D	0212	Y
04	285	V	TETRACHLOROETHENE-1,2-13C2	D	0172	Y
04	286	V	TOLUENE-2,3,4,5,6-D8	D	0098	Y
04	287	V	TRICHLOROETHENE-13C2	D	0136	Y
04	288	V	VINYL CHLORIDE-D3	D	0065	Y
04	289	P	ALDRIN-13C4	D		N
04	290	P	DIELDRIN-13C4	D		N
04	291	P		D		N
04	292	P	4,4'-DDT-D8	D		N
04	293	P	4,4'-DDE-D8	D		N
04	294	P	4,4'-DDD-D8	D		N
04	295	P	ENDOSULFAN-I-D4	D		N
04	296	P		D		N

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TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	297	P		D		N
04	298	P		D		N
04	299	P		D		N
04	301	B	ACENAPHTHENE	P	0154	Y
04	302	V	ACROLEIY	P	0056	Y
04	303	V	ACRYLONITRILE	P	0053	Y
04	304	V	BENZENE	P	0078	Y
04	305	B	BENZIDINE	P	0184	Y
04	306	V	CARBON TETRACHLORIDE	P	0047	Y
04	307	V	CHLOROBENZENE	P	0112	Y
04	308	B	1,2,4-TRICHLOROBENZENE	P	0180	Y
04	309	B	HEXACHLOROBENZENE	P	0284	Y
04	310	V	1,2-DICHLOROETHANE	P	0062	Y
04	311	V	1,1,1-TRICHLOROETHANE	P	0097	Y
04	312	B	HEXACHLOROETHANE	P	0201	Y
04	313	V	1,1-DICHLOROETHANE	P	0063	Y
04	314	V	1,1,2-TRICHLOROETHANE	P	0083	Y
04	315	V	1,1,2,2-TETRACHLOROETHANE	P	0083	Y
04	316	V	CHLOROETHANE	P	0064	Y
04	317	V		P		N
04	318	B	BIS(2-CHLOROETHYL)ETHER	P	0093	Y
04	319	V		P	0106	N
04	320	B	2-CHLORONAPHTHALENE	P	0162	Y
04	321	A	2,4,6-TRICHLOROPHENOL	P	0196	Y
04	322	A	4-CHLORO-3-METHYLPHENOL	P	0107	Y
04	323	V	CHLOROFORM	P	0085	Y
04	324	A	2-CHLOROPHENOL	P	0128	Y
04	325	B	1,2-DICHLOROBENZENE	P	0146	Y
04	326	B	1,3-DICHLOROBENZENE	P	0146	Y
04	327	B	1,4-DICHLOROBENZENE	P	0146	Y
04	328	B	3,3'-DICHLOROBENZIDINE	P	0252	Y
04	329	V	1,1-DICHLOROETHENE	P	0061	Y
04	330	V	TRANS-1,2-DICHLOROETHENE	P	0061	Y
04	331	A	2,4-DICHLOROPHENOL	P	0162	Y
04	332	V	1,2-DICHLOROPROPANE	P	0063	Y
04	333	V	T-1,3-DICHLOROPROPENE	P	0075	Y
04	334	B	2,4-DIMETHYLPHENOL	P	0122	Y
04	335	B	2,4-DINITROTOLUENE	P	0165	Y
04	336	B	2,6-DINITROTOLUENE	P	0165	Y
04	337	B	1,2-DIPHENYLHYDRAZINE	P	0077	Y
04	338	V	ETHYLBENZENE	P	0106	Y
04	339	B	FLUORANTHENE	P	0202	Y
04	340	B	4-CHLOROPHENYL PHENYL ETHER	P	0204	Y
04	341	B		P		N
04	342	B	BIS (2-CHLOROISOPROPYL) ETHER	P	0121	Y
04	343	B		P		N
04	344	V	METHYLENE CHLORIDE	P	0084	Y
04	345	V	CHLOROMETHANE	P	0050	Y
04	346	V	BROMOMETHANE	P	0096	Y
04	347	V	BROMOFORM	P	0173	Y

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TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	348	V	BROMODICHLOROMETHANE	P	0083	Y
04	349	V		P		N
04	350	V		P		N
04	351	V	DIBROMOCHLOROMETHANE	P	0129	Y
04	352	B	HEXACHLORO-1,3-BUTADIENE	P	0225	Y
04	353	B	HEXACHLOROCYCLOPENTADIENE	P	0237	Y
04	354	B	ISOPHORONE	P	0082	Y
04	355	B	NAPHTHALENE	P	0128	Y
04	356	B	NITROBENZENE	P	0123	Y
04	357	A	2-NITROPHENOL	P	0139	Y
04	358	A	4-NITROPHENOL	P	0139	Y
04	359	A	2,4-DINITROPHENOL	P	0184	Y
04	360	A	2-METHYL-4,6-DINITROPHENOL	P	0198	Y
04	361	B		P		N
04	362	B	N-NITROSODIPHENYLAMINE	P	0169	Y
04	363	B		P		N
04	364	A	PENTACHLOROPHENOL	P	0266	Y
04	365	B	PHENOL	P	0094	Y
04	366	B	BIS (2-ETHYLHEXYL) PHTHALATE	P	0149	Y
04	367	B		P		N
04	368	B	DI-N-BUTYL PHTHALATE	P	0149	Y
04	369	B	DI-N-OCTYL PHTHALATE	P	0149	Y
04	370	B	DIETHYL PHTHALATE	P	0149	Y
04	371	B	DIMETHYL PHTHALATE	P	0163	Y
04	372	B	BENZO(A)ANTHRACENE	P	0228	Y
04	373	B	BENZO(A)PYRENE	P	0252	Y
04	374	B	BENZO(B)FLUORANTHENE	P	0252	Y
04	375	B	BENZO(K)FLUORANTHENE	P	0252	Y
04	376	B	CHRYSENE	P	0228	Y
04	377	B	ACENAPHTHYLENE	P	0152	Y
04	378	B	ANTHRACENE	P	0178	Y
04	379	B	BENZO(GHI)PERYLENE	P	0276	Y
04	380	B	FLUORENE	P	0166	Y
04	381	B	PHENANTHRENE	P	0178	Y
04	382	B		P		N
04	383	B		P		N
04	384	B	PYRENE	P	0202	Y
04	385	V	TETRACHLOROETHENE	P	0166	Y
04	386	V	TOLUENE	P	0092	Y
04	387	V	TRICHLOROETHENE	P	0095	Y
04	388	V	VINYL CHLORIDE	P	0062	Y
04	389	P		P		N
04	390	P		P		N
04	391	P		P		N
04	392	P		P		N
04	393	P		P		N
04	394	P		P		N
04	395	P		P		N
04	396	P		P		N
04	397	P		P		N

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TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	398	P		P		N
04	399	P		P		N
04	400	P	HEPATACHLOR-13C4	D		N
04	402	P	ALPHA-BHC-D6	D		N
04	404	P	GAMMA-BHC-D6	D		N
04	429	D	2,3,7,8-TCDD-13C12	D	0332	Y
04	500	A	BENZOIC ACID	P		Y
04	501	A	HEXANOIC ACID	P		Y
04	502	B	B-NAPHTHYLAMINE	P	0143	Y
04	503	B	ALPHA-PICOLINE	P	0093	Y
04	504	B	DIBENZOTHIOPHENE	P	0184	Y
04	505	B	DIBENZOFURAN	P	0168	Y
04	506	B	N-DODECANE (N-C12)	P	0055	Y
04	507	B	DIPHENYLAMINE	P	0169	Y
04	508	B	DIPHENYL ETHER	P	0170	Y
04	509	B	ALPHA-TERPINEOL	P	0059	Y
04	510	B	STYRENE	P	0104	Y
04	511	B	DI-N-BUTYL AMINE	P	0086	N
04	512	B	BIPHENYL	P	0154	Y
04	513	B	P-CYMEME	P	0119	Y
04	514	V	2-BUTANONE (MEK)	P	0072	Y
04	515	V	DIETHYL ETHER	P	0074	Y
04	516	V	ACETONE	P	0058	Y
04	517	B	N-DECANE (N-C10)	P	0055	Y
04	518	B	N-TETRADECANE (N-C14)	P	0055	Y
04	519	B	N-HEXADECANE (N-C16)	P	0055	Y
04	520	B	N-OCTADECANE (N-C18)	P	0055	Y
04	521	B	N-EICOSANE (N-C20)	P	0055	Y
04	522	B	N-DOCOSANE (N-C22)	P	0055	Y
04	523	B	N-TETRACOSANE (N-C24)	P	0055	Y
04	524	B	N-HEXACOSANE (N-C26)	P	0055	Y
04	525	B	N-OCTACOSANE (N-C28)	P	0055	Y
04	526	B	N-TRIACONTANE (N-C30)	P	0055	Y
04	527	V	P-DIOXANE	P	0088	Y
04	528	B	CARBAZOLE	P	0167	Y
04	529	B	1,2,3-TRICHLOROBENZENE	P	0180	Y
04	530	A	2,3,6-TRICHLOROPHENOL	P	0196	Y
04	531	A	2,4,5-TRICHLOROPHENOL	P	0196	Y
04	532	V	ALLYL ALCOHOL	P	0057	Y
04	533	V	CARBON DISULFIDE	P	0076	Y
04	534	V	2-CHLORO-1,3-BUTADIENE	P	0053	Y
04	535	V	CHLOROACETONITRILE	P	0075	Y
04	536	V	3-CHLOROPROPENE	P	0076	Y
04	537	V	CROTONALDEHYDE	P	0070	Y
04	538	V	1,2-DIBROMOETHANE (EDB)	P	0107	Y
04	539	V	DIBROMOMETHANE	P	0093	Y
04	540	V	TRANS-1,4-DICHLORO-2-BUTENE	P	0075	Y
04	541	V	1,3-DICHLOROPROPANE	P	0076	Y
04	542	V	CIS-1,3-DICHLOROPROPENE	P	0075	Y
04	543	V	ETHYL CYANIDE	P	0054	Y

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             COMPOUND CODE TABLE

 TABLE        TABLE                                        CODE
 NUMBER        CODE                                    DESCRIPTION

   04        544            V ETHYL METHACRYLATE                P 0069 Y
   04        545            V 2-HEXANONE                        P 0058 Y
   04        546            V IODOMETHANE                       P 0142 Y
   04        547            V ISOBUTYL ALCOHOL                  P 0074 Y
   04        548            V METHACRYLONITRILE                 P 0067 Y
   04        549            V METHYL METHACRYLATE               P 0069 Y
   04        550            V 4-METHLY-2-PENTANONE              P 0058 Y
   04        551            V 1,1,1,2-TETRACHLOROETHANE         P 0131 Y
   04        552            V TRICHLOROFLUOROMETHANE            P 0101 Y
   04        553            V 1,2,3-TRICHLOROPROPANE            P 0075 Y
   04        554            V VINYL ACETATE                     P 0086 Y
   04        555            B ACETOPHENONE                      P 0105 Y
   04        556            B 4-AMINOBIPHENYL                   P 0169 Y
   04        557            B ANILINE                           P 0093 Y
   04        558            B O-ANISIDINE                       P 0108 Y
   04        559            B ARAMITE                           P 0185 Y
   04        560            B BENZANTHRONE                      P 0230 Y
   04        561            B 1,3-BENZENEDIOL (RESORCINOL)      P 0110 Y
   04        562            B BENZENETHIOL                      P 0110 Y
   04        563            B 2,3-BENZOFLUORENE                 P 0216 Y
   04        564            B BENZYL ALCOHOL                    P 0079 Y
   04        565            B 2-BROMOCHLOROBENZENE              P 0111 Y
   04        566            B 3-BROMOCHLOROBENZENE              P 0192 Y
   04        567            B 4-CHLORO-2-NITROANILINE           P 0172 Y
   04        568            B 5-CHLORO-O-TOLUIDINE              P 0106 Y
   04        569            B 4-CHLOROANILINE                   P 0127 Y
   04        570            B 3-CHLORONITROBENZENE              P 0157 Y
   04        571            B O-CRESOL                          P 0108 Y
   04        572            B CROTOXYPHOS                       P 0127 Y
   04        573            B 2,6-DI-TERT-BUTYL-P-BENZOQINO     P 0220 Y
   04        574            B 2,4-DIAMINOTOLUENE                P 0122 Y
   04        575            B 1,2-DIBROMO-3-CHLOROPROPANE       P 0157 Y
   04        576            B 2,6-DICHLORO-4-NITROANILINE       P 0124 Y
   04        577            B 1,3-DICHLORO-2-PROPANOL           P 0079 Y
   04        578            B 2,3-DICHLOROANILINE               P 0161 Y
   04        579            B 2,3-DICHLORONITROBENZENE          P 0191 Y
   04        580            B 1,2:3,4-DIEPOXYBUTANE             P 0055 Y
   04        581            B 3,3'-DIMETHOXYBENZIDINE           P 0224 Y
   04        582            B DIMETHYL SULFONE                  P 0079 Y
   04        583            B P-DIMETHYLAMINOAZOBENZENE         P 0120 Y
   04        584            B 7,12-DIMETHYLBENZ(A)ANTHRACENE    P 0256 Y
   04        585            B N,N-DIMETHYLFORMAMIDE             P 0073 Y
   04        586            B 3,6-DIMETHYLPHENANTHRENE          P 0206 Y
   04        587            B 1,3-DINITROBENZENE                P 0168 Y
   04        588            B DIPHENYLDISULFIDE                 P 0218 Y
   04        589            B ETHYL METHANESULFONATE            P 0109 Y
   04        590            B ETHYLENETHIOUREA                  P 0102 Y
   04        591            B ETHYNYLESTRADIOL 3-METHYL ETHE    P 0227 Y
   04        592            B HEXACHLOROPROPENE                 P 0213 Y
   04        593            B 2-ISOPROPYLNAPHTHALENE            P 0170 Y
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COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	594	B	ISOSAFROLE	P	0162	Y
04	595	B	LONGIFOLENE	P	0161	Y
04	596	B	MALACHITE GREEN	P	0330	Y
04	597	B	METHAPYRILENE	P	0097	Y
04	598	B	METHYL METHANESULFONATE	P	0080	Y
04	599	B	2-METHYLBENZOTHIOAZOLE	P	0149	Y
04	600	A	BENZOIC-D5 ACID	D		N
04	601	A	HEXANOIC ACID-D11	D		N
04	602	B	B-NAPHTHYLAMINE-D7	D	0150	Y
04	603	B	ALPHA-PICOLINE-D7	D	0100	Y
04	604	B	DIBENZOTHIOPHENE-D8	D	0192	Y
04	605	B	DIBENZOFURAN-D8	D	0176	Y
04	606	B	N-DODECANE-D26 (N-C12)	D	0066	Y
04	607	B	DIPHENYLAMINE-D10	D	0179	Y
04	608	B	DIPHENYL ETHER-D10	D	0180	Y
04	609	B	ALPHA-TERPINEOL-D3	D	0062	Y
04	610	B	STYRENE-2,3,4,5,6-D5	D	0109	Y
04	611	B	DI-N-BUTYL AMINE-D18	D	0096	N
04	612	B	BIPHENYL-D10	D	0164	Y
04	613	B	P-CYMENE-D14	D	0130	Y
04	614	V	2-BUTANONE-4,4,4-D3 (MEK)	D	0075	Y
04	615	V	DIETHYL ETHER-D10	D	0084	Y
04	616	V	ACETONE-D6	D	0064	Y
04	617	B	N-DECANE-D22 (N-C10)	D	0066	Y
04	618	B		D		N
04	619	B	N-HEXADECANE-D34 (N-C16)	D	0066	Y
04	620	B		D		N
04	621	B	N-EICOSANE-D42 (N-C20)	D	0066	Y
04	622	B		D		N
04	623	B	N-TETRACOSANE-D50 (N-C24)	D	0066	Y
04	624	B		D		N
04	625	B		D		N
04	626	B	N-TRIACONTANE-D62 (N-C30)	D	0066	Y
04	627	V	P-DIOXANE D8	D	0096	N
04	628	B	CARBAZOLE D8	D	0175	N
04	629	B	1,2,3-TRICHLOROBENZENE D3	D	0183	N
04	630	A	2,3,6-TRICHLOROPHENOL D2	D	0200	N
04	631	A	2,4,5-TRICHLOROPHENOL D2	D	0200	N
04	700	A	BENZOIC ACID	P		Y
04	701	A	HEXANOIC ACID	P		Y
04	702	B	B-NAPHTHYLAMINE	P	0143	Y
04	703	B	2-METHYLPYRIDINE	P	0093	Y
04	704	B	DIBENZOTHIOPHENE	P	0184	Y
04	705	B	DIBENZOFURAN	P	0168	Y
04	706	B	N-DODECANE (N-C12)	P	0055	Y
04	707	B	DIPHENYLAMINE	P	0169	Y
04	708	B	DIPHENYL ETHER	P	0170	Y
04	709	B	ALPHA-TERPINEOL	P	0059	Y
04	710	B	STYRENE	P	0104	Y
04	711	B	DI-N-BUTYL AMINE	P	0086	N

COMPOUND CODE TABLE

TABLE NUMBER	TABLE CODE		CODE DESCRIPTION			
04	712	B	BIPHENYL	P	0154	Y
04	713	B	P-CYMENE	P	0119	Y
04	714	V	2-BUTANONE (MEK)	P	0072	Y
04	715	V	DIETHYL ETHER	P	0074	Y
04	716	V	ACETONE	P	0058	Y
04	717	B	N-DECANE (N-C10)	P	0055	Y
04	718	B		P		N
04	719	B	N-HEXADECANE (N-C16)	P	0055	Y
04	720	B		P		N
04	721	B	N-EICOSANE (N-C20)	P	0055	Y
04	722	B		P		N
04	723	B	N-TETRACOSANE (N-C24)	P	0055	Y
04	724	B		P		N
04	725	B		P		N
04	726	B	N-TRIACONTANE (N-C30)	P	0055	Y
04	727	V	P-DIOXANE	P	0088	N
04	728	B	CARBAZOLE	P	0167	N
04	729	B	1,2,3-TRICHLOROBENZENE	P	0180	N
04	730	A	2,3,6-TRICHLOROPHENOL	P	0196	N
04	731	A	2,4,5-TRICHLOROPHENOL	P	0196	N
04	829	D	2,3,7,8-TCDD	P	0320	Y
04	900	B	3-METHYLCHOLANTHRENE	P	0268	Y
04	901	B	4,4'-METHYLENEBIS(2-CHLOROANI)	P	0231	Y
04	902	B	4,5-METHYLENEPHENANTHRENE	P	0190	Y
04	903	B	1-METHYLFLUORENE	P	0180	Y
04	904	B	2-METHYLNAPHTHALENE	P	0142	Y
04	905	B	1-METHYLPHENANTHRENE	P	0192	Y
04	906	B	2-(METHYLTHIO)BENZOTHIAZOLE	P	0181	Y
04	907	B	1,5-NAPHTHALENEDIAMINE	P	0158	Y
04	908	B	1,4-NAPHTHOQUINONE	P	0158	Y
04	909	B	ALPHA-NAPHTHYLAMINE	P	0143	Y
04	910	B	5-NITRO-O-TOLUIDINE	P	0152	Y
04	911	B	2-NITROANILINE	P	0138	Y
04	912	B	3-NITROANILINE	P	0138	Y
04	913	B	4-NITROANILINE	P	0138	Y
04	914	B	4-NITROBIPHENYL	P	0199	Y
04	915	B	N-NITROSODI-N-BUTYLAMINE	P	0084	Y
04	916	B	N-NITROSODIETHYLAMINE	P	0102	Y
04	917	B	N-NITROSOMETHYLETHYLAMINE	P	0088	Y
04	918	B	N-NITROSOMETHYLPHENYLAMINE	P	0106	Y
04	919	B	N-NITROSOMORPHOLINE	P	0056	Y
04	920	B	N-NITROSOPIPERIDINE	P	0114	Y
04	921	B	PENTACHLOROBENZENE	P	0248	Y
04	922	B	PENTACHLOROETHANE	P	0117	Y
04	923	B	PENTAMETHYLBENZENE	P	0148	Y
04	924	B	PERYLENE	P	0252	Y
04	925	B	PHENACETIN	P	0108	Y
04	926	B	PHENOTHIAZINE	P	0199	Y
04	927	B	1-PHENYLNAPHTHALENE	P	0204	Y
04	928	B	2-PHENYLNAPHTHALENE	P	0204	Y

```
10/30/86                    I S O T O P E   D I L U T I O N

          COMPOUND CODE TABLE

TABLE     TABLE                                       CODE
NUMBER     CODE                                   DESCRIPTION

  04      929          B PRONAMIDE                          P 0173 Y
  04      930          B PYRIDINE                           P 0079 Y
  04      931          B SAFROLE                            P 0162 Y
  04      932          B SQUALENE                           P 0069 Y
  04      933          B 1,2,4,5-TETRACHLOROBENZENE         P 0216 Y
  04      934          B THIANAPHTHENE                      P 0134 Y
  04      935          B THIOACETAMIDE                      P 0075 Y
  04      936          B THIOXANTHONE                       P 0212 Y
  04      937          B O-TOLUIDINE                        P 0106 Y
  04      938          B 1,2,3-TRIMETHOXYBENZENE            P 0168 Y
  04      939          B 2,4,5-TRIMETHYLANILINE             P 0120 Y
  04      940          B TRIPHENYLENE                       P 0228 Y
  04      941          B TRIPROPYLENEGLYCOL METHYL ETHE P 0059 Y
  04      942          B 1,3,5-TRITHIANE                    P 0138 Y
  04      943          A BENZOIC ACID                       P 0105 Y
  04      944          A P-CRESOL                           P 0108 Y
  04      945          A 3,5-DIBROMO-4-HYDROXYBENZONITR P 0227 Y
  04      946          A 2,6-DICHLOROPHENOL                 P 0166 Y
  04      947          A HEXANOIC ACID                      P 0060 Y
  04      948          A 2,3,4,6-TETRACHLOROPHENOL          P 0232 Y

    TABLE COUNT:      570
```

ATTACHMENT 10

APPENDIX G

STORAGE, PREPARATION AND SHIPMENT OF SAMPLE EXTRACTS

APPENDIX G

STORAGE, PREPARATION AND SHIPMENT OF SAMPLE EXTRACTS

A. Storage

1. All samples/sample extracts shall be stored in a dark contaminant-free area at −20 to −10°C, except when samples/sample extracts are being analyzed. A line shall be marked on sample containers at the solvent level, per the method. Extracts shall be tightly capped with unpierced septa or cap liners to prevent sample loss by evaporation.

B. Preparation

1. Contractor shall label each extract with the EPA Episode and Sample (Traffic Report) number, in addition the information on the Sample Extract Transmittal Form (Figure 1) shall be completed in full and signed by the appropriate persons. EPA Episode and Sample numbers on the extract labels shall agree with the numbers on the "Transmittal" form.

2. Sample containers shall be packed in foam or other material which will preclude breakage during shipment. Packaging in "Vial File" or other compartmentalized container is recommended. NOTE: If breakage occurs during shipment, and it is determined that such breakage is due to improper packaging on the part of the Contractor, the Contractor shall be subject to liquidated damages as specified in the contract.

C. Shipment

1. Contractor shall ship sample extracts via approved ground carrier (i.e. UPS or equivalent) in consideration of solvents used in extraction. All containers in the shipment shall be marked according to applicable DOT regulations for the concerned compounds.

2. Contractor shall ship sealed vials containing labeled extracts and the applicable "Sample Extract Transmittal Form" to:

AquAir Corporation
13300 Sam Neely Drive
P.O. Box 7048
Charlotte, North Carolina 28217

Attn: James H. Pugh

USEPA Industrial Technology Division
Sample Extract Transmittal Form

Revised October 1986

Episode Number	Industrial Category	Sample Number		Extraction		Conc factor	Dates			Int std		Initials	
		EPA	Lab	Frac	Type		Rec	Extr	Analy	Type	Conc*	Spec	QA

The extracts in this shipment were estracted and concentrated per all contract specifications.

Date: ____________ Extraction Specialist: ____________ QA: ____________

ATTACHMENT 11

APPENDIX F

GC/MS RAW DATA ON MAGNETIC TAPE

APPENDIX F
GC/MS RAW DATA ON MAGNETIC TAPE

A. All GC/MS mass intensity data from analyses of samples, sample dilutions, blanks, calibration standards, initial and ongoing precision and recovery, and BFB and DFTPP shall be reporded on magnetic tape per the specifications below:

1. Data Format

Type:	9-track BPI, 2,400 foot reels
Record Length:	80
Block Size	4,000 (specify)
Code:	ASCII

2. The first two (2) records shall contain the sample identification and other information necessary to rigorously identify the sample. Subsequent records shall contain eight (8) mass-intensity pairs, each of which shall be ten (10) characters long. Each mass and each intensity shall be five (5) characters long and left justified. At the end of each spectrum in a single run, the last mass-intensity pair shall be "zero Zero" (00) to denote the end of the spectrum. An end-of-file mark shall be placed at the end of each GC/MS run. For example:

2 Records	Sample 1 identification
N Records:	Spectrum 1 of sample, last mass-intensity pair is "00" to denote end of spectrum
M Records:	Spectrum 2 of sample, last mass intensity pair is "00" to denote end of spectrum
End of File	
2 Records:	Sample 2 identification
	... etc ...

3. Contractor shall package magnetic tapes carefully to ensure that they are not damaged in shipment and ship them by first class mail or equivalent.

   Contractor shall complete a GC/MS Raw Data Tape Transmittal Form for each magnetic tape submitted.

   Contractor shall ship magnetic tapes containing GC/MS raw data and the applicable GC/MS Raw Data Transmittal Form to:

   ITD Sample Control Center
   300 N. Lee Street, Suite 200
   Alexandria, Virginia 22313

USEPA Industrial Technology Division
GCMS Raw Data Tape Transmittal

Revised October 1986

Episode	Industrial	Sample Number			Analysis		Date	Initials	
Number	Category	EPA	Lab	Data File Name	Frac	Type	Analyzed	Analyst	QA

The data recorded on this tape has been verified and meets all contract specifications.

Date: ____________ Analyst: ____________ QA: ____________

ATTACHMENT 12

EPISODE NO.	
SAMPLE NO.	

LABORATORY RESULTS

METALS ANALYSIS

Lab Name____________________ Sample Point____________________

Industrial Category____________________ Sample Date____________________

Date Analyzed____________________ Standard____________________

ICP RESULTS

		ug/l			ug/l
1.	Calcium		11.	Cadmium	
2.	Magnesium		12.	Molybdenum	
3.	Sodium		13.	Tin	
4.	Aluminum		14.	Yttrium	
5.	Manganese		15.	Cobalt	
6.	Lead		16.	Chromium	
7.	Vanadium		17.	Copper	
8.	Boron		18.	Iron	
9.	Barium		19.	Nickel	
10.	Beryllium		20.	Titanium	
			21.	Zinc	

FURNACE AA RESULTS

			ug/l				ug/l
___	1.	Selenium		___	4.	Arsenic	
___	2.	Thallium		___	5.	Antimony	
___	3.	Silver		___	6.	Mercury	

COMMENTS:

ATTACHMENT 13

SCC TAPE PROCESSING STEPS

STEP 1. DEBLOCK tape.

STEP 2. REVIEW Lab Transmittal/Error Report.

STEP 3. ACCEPT or REJECT Tape:
~ REJECTED -- return Tapes to Lab.
~ ACCEPTED -- Continue to STEP 4.

STEP 4. ARCHIVE Original Quantitation Reports received.

STEP 5. Make Quantitation Report Corrections as required:
~ POST CHANGES in individual Quantitation Reports.
~ RERUN Lab Transmittal/Error Report.
~ REPEAT UNTIL NO ERRORS.

STEP 6. Run Tracking File Merge.

STEP 7. Run LEVEL 1 Edits on Data:
~ VERIFY all Edit Runs to insure successful completion.
~ RESOLVE Processing Problems with DP.

STEP 8. Run LEVEL 1 Error Summary Report.

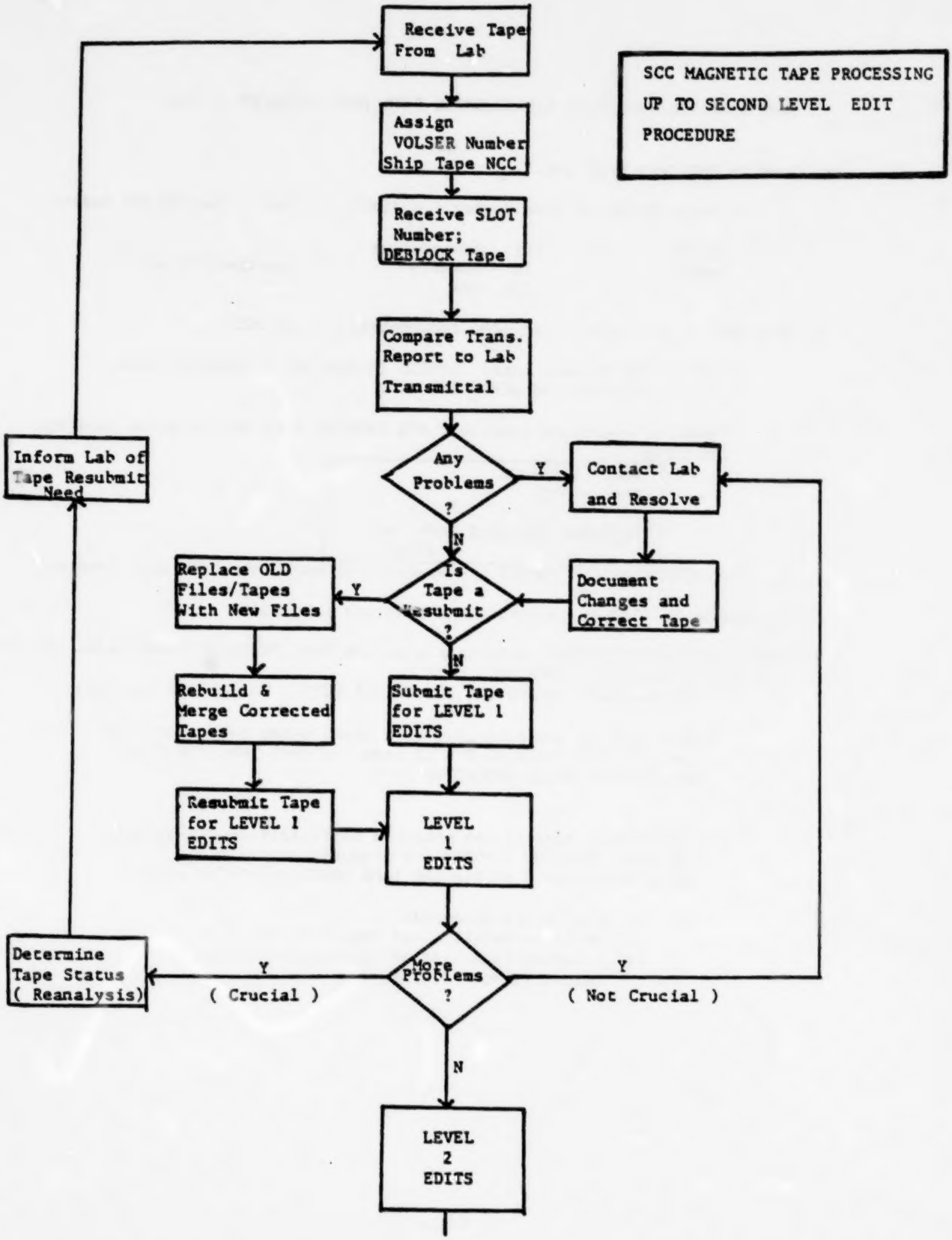
STEP 9. Run LEVEL 2 Edits:
~ VERIFY all Edit Runs to insure successful completion.
~ RESOLVE Processing Problems with DP.

STEP 10. Make LEVEL 1 Edits, LEVEL 1 Error Summary Report and LEVEL 2 Edits available for QC Review.

STEP 11. Results of QC Review:
~ TOTAL NON-COMPLIANCE
  (1) Return Tapes to Lab.
  (2) Retain original archived Quantitation Reports until data is resubmitted. When received, replace with new submission.
~ INDIVIDUAL QUANTITATION CORRECTIONS
  (1) Make corrections to individual Quantitations.
  (2) Changes to header information must be coordinated with DP.
  (3) Repeat procedure from STEP 5.
~ TOTAL COMPLIANCE Continue with STEP 12.

STEP 12. Run Remedial Report and make available to EGD.

STEP 13. ARCHIVE Modified Quantitation Files (P010).



SCC MAGNETIC TAPE PROCESSING UP TO SECOND LEVEL EDIT PROCEDURE

Receive Tape From Lab
Assign VOLSER Number Ship Tape NCC
Receive SLOT Number; DEBLOCK Tape
Compare Trans. Report to Lab Transmittal
Any Problems ?
Y
Contact Lab and Resolve
Document Changes and Correct Tape
N
Is Tape a Resubmit ?
Y
Replace OLD Files/Tapes With New Files
Rebuild & Merge Corrected Tapes
Resubmit Tape for LEVEL 1 EDITS
N
Submit Tape for LEVEL 1 EDITS
LEVEL 1 EDITS
More Problems ?
Y
(Crucial)
Determine Tape Status (Reanalysis)
Inform Lab of Tape Resubmit Need
Y
(Not Crucial)
N
LEVEL 2 EDITS

11-30-87

VIAR HANDLING PROCEDURE FOR INCOMING DATA TAPE PACKAGES -- SCC

1. TAPE IS RECEIVED FROM LAB.

   " Assign External Tape Number (6 digit); also called VOLSER Number

VOLSER Number	--->	lab ID	consecutive Number	<< Lab-Specific >>
		XXX	XXX	

2. SEND TAPE (with VIAR - assigned documentation) to NCC.

   " Affix TAG to each tape: EPATQS (S.Hancock),EGLDSSCSP,C816, (703) 557-5040, VOLSER = XXX XXX.

   " Send by overnight courier (FED EXPRESS) to NCC in North Carolina ADDRESS:

   TAPE LIBRARIAN
   EPA -- NCC
   MD - 24
   RESEARCH TRIANGLE PARK, NC
   27711

3. NCC WILL ASSIGN A SLOT NUMBER (8 Number) to each tape, and will transmit

   This Number to VIAR by telephone the next day (by 12:00)

4. TAPE DEBLOCKING PROCESS (Selection 1 on LAB TAPE TRACKING SYSTEM menu (Selection 4 on SAMPLE CONTROL CENTER MAIN MENU)

   " Use the SLOT NUMBER and the VOLSER NUMBER to Deblock the Tape.

   " Only 8 files are deblocked at a time, using Selection 1 on the LAB TAPE TRACKING SYSTEM menu << TAPE TRACKING FILE DEBLOCK AND BUILD PROCEDURE >>

   " Transmittal Reports are produced as deblocking job output. LAB TAPE TRACKING REPORTS are produced separately, using Selection 5 on the LAB TAPE TRACKING SYSTEM menu.

   " Two types of files produced:
     (1) multiple quantitative report files
     (2) a master file/list of these quantitative report files (CONTROL)

5. Quantitative Report Corrections are made.

- Contact lab for solving discrepancies if needed.
- Make necessary changes to Header Information
- Compare tape transmittal form (Master List from tape) with hard copy transmittal forms and resolve all discrepancies.
- Can't do Level 1 Edits until header information corrected.

- Check DATE/TIME
- Check for duplicate compound entries

6. DO LEVEL 1 EDITS

- If the Tape is a NEW TAPE, and not a resubmittal tape (to correct erroneous files in previously submitted tapes), the next step is to submit the tape for LEVEL 1 EDITS.
- If the Tape is a resubmittal, see Procedure 7.
- When scanning the LAB TAPE TRACKING REPORT, produced by Selection 5 on the LAB TAPE TRACKING SYSTEM main menu, a 'G' indicates a no-error status.

- Level 1 Edits of disk quantitation files are performed:
  (1) tape specs verified
  (2) data completeness checked
  (3) retention times, RRTs checked
- If no errors detected, data from disk quantitation files are merged with output data set. (Selection 3, LAB TAPE TRACKING SYSTEM MENU << TAPE TRACKING FILE MERGE PROCEDURE >>)

- Level 2 Edits may be performed.
- Level 1 Summary Error Listing may be produced if desired.
- If REJECTED, tapes are returned to lab, and corrected tape is resumbitted.

7. TAPE RESUBMISSION PROCEDURE.

- After the RESUBMISSION TAPE is Deblocked, the old files (from the tapes in error) are replaced with corrected files from the replacement tape, Selection 5 on the QUANTITATION REPORT CORRECTION PROCESSING menu, << ' Quantitation Report Processing; Quantitation File Replacement Procedure ' >>

- After replacing the erroneous files with the correct files from the new tape, the corrected tapes are

  Rebuilt -- Selection 2 on the LAB TAPE TRACKING SYSTEM Menu; then

  Merged -- Selection 3 on the LAB TAPE TRACKING SYSTEM Menu.

- Corrected Tapes can then be resubmitted for LEVEL 1 Edits, Selection 2 on the LABORATORY PROCESSING Menu, ' Resubmit Disk Quantitation Files '. Files are edited one file a time.

- Only 1 file at a time is edited, except for ASSOCIATED files (have same ID, done at same time and date)

ATTACHMENT 14

QUALITY ASSURANCE TESTS OF DATA ELEMENTS ON QUANTITATION REPORTS

This document describes tests of data fields which occur when a gas chromatography-mass spectrometry (GCMS) quantitation report is processed through EPA's computerized quality assurance system at the Agency's National Computer Center (NCC). GCMS quantitation reports are received on magnetic tape from laboratories performing chemical analyses of organic pollutants. The specifications for these quantitation reports are contained in an EPA document titled "Quantitation Reports on Magnetic Tape." The data elements tested are defined in an EPA document titled "EGD Data Elements," and an outline of the tape processing steps is given in an EPA Sample Control Center document titled "SCC Tape Processing Steps."

1. General Tests

   1.1 Multiple header records (type 10) may be present. If present, the records must be equal. If not equal, an error condition exists.

   1.2 There must be only one compound record for each compound analyzed. Duplicate records for a given compound are prohibited with one exception: duplicate records for internal standards. With this exception, duplicate records constitute an error condition.

   1.3 Multiple compound records for internal standards may be present. If present, all records for each internal standard must be equal. If not equal, an error condition exists.

   1.4 All isotope compounds (200 and 600 series) for the fraction analyzed must be present in the quan report. If any are missing, an error condition exists.

   1.5 For semi-volatiles, the only valid internal standard is compound

164 (2,2'-difluorobiphenyl). For volatiles, the only valid internal standards are compounds 181, 182, and 183 (bromochloromethane; 2-bromo, 1-chloropropane; and 1,4-dichlorobutane). Each of these three must be present in the quan report for volatiles.

1.6 For quan reports submitted in multiple parts (termed "Associated Quans"), there must be identical internal standard compounds present in each part. The number of identical header records shall be used to establish the number of parts present. If the multiple internal standard records are not present, an error condition exists.

1.7 There must be at least one header record present for a quantitation report. If no header record is present, an error condition exists.

2. Processing of "Not Found" compounds

2.1 The following fields must be present for a compound not found:

CMPD Number
CMPD Order Number
M/Z
REF CMPD Number
RT (LIB)
RRT (LIB)
AMT (LIB)
R. FAC (LIB)

The above fields will be edited.

2.2 The following fields must not be present. If present, an error condition exists:

SCAN
RT

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RRT

AREA

AMOUNT

UNITS of Measure

R. FAC

2.3 The above fields will not be edited.

2.4 A Not Found condition is determined all above fields not being present.

3. Detailed Tests--the following table summarizes the data fields tested. The numbers are used to reference the detailed tests described below:

Data Field	Field Number
Amount	26
Amount (Library)	30
Bottle Number	7
Carrier Gas Flow Rate	17
Column Final Hold	16
Column Initial Hold	14
Column Inside Diameter	13
Column Length	12
Column Temperature Program	15
Compound Number	19
Compound Reference Number	18
Concentration/Dilution Factor	9
Date Analyzed	1
Date Extracted	10
Fraction	8

Instrument	3
Mass to Charge Ratio	20
Method	11
Peak Area	25
Quantitation Report Type	5
Reference Compound	23
Relative Retention Time	24
Relative Retention Time (Library)	29
Response Factor	31
Response Factor (Library)	28
Sample Number	6
Scan Number	21
Analysis Shift	4
Time Analyzed	2
Unit of Measure	27

1 Date Analyzed

1.1 Must be present

1.2 Format: MM/DD/YY

1.3 Limits: MM=01-12 DD=01-31 YY=83-99

2 Time Analyzed

2.1 Must be present

2.2 Format: HH:MM:SS or H:MM:SS or MM:SS

2.3 Limits: HH=00-24 MM=00-59 SS=00-59

3 Instrument

3.1 Must be present

3.2 Must be a valid instrument code for the lab (Table 11)

4 Shift

4.1 Must be present

4.2 Must be a valid code: D=Day S=Swing G=Graveyard

5 Quan Report Type

5.1 Must be present

5.2 Must be a valid code (Table 12)

5.3 Must be valid for Method (Table 12)

6 Sample Number

6.1 Must be present

6.2 For quan report type EPA:

6.2.1 Must be valid number in Episode/Sample File

6.3 For quan report type CAL or VER:

6.3.1 Must be valid concentration: 00010; 00020; 00050; 00100; 00200 (or 5x these amounts for volatile gases and water soluble compounds).

6.4 For quan report type BLK:

6.4.1 Must be 00000

6.5 For remaining quan report types: must be numeric

7 Bottle Number

7.1 Must be present

7.2 For quan report type EPA:

7.2.1 Must be in range of 00 to 99

7.2.2 For TCLP: must be 99

7.3 For remaining quan report types: must be 00

8 Fraction

8.1 Must be present

8.2 Must be a valid code: A, B, C, P, V

8.3 Must be valid for the method and quan report type

8.3.1 For 1624: V only

8.3.2 For 1625, type EPA: A, B, C, P

8.3.3 For 1625, all others: C

9 Concentration/dilution factor

9.1 Must be present

9.2 Format: XXXXX:XXXXX

9.3 For volatile fraction (V):

9.3.1 For aqueous samples: must be 1:XXXXX

9.3.1.1 For quan types CAL, APS, VER, BLK, must be 1:1

9.3.2 For solid samples: must be S(.)N(.)N(.)N:XXXXX, where S indicates a solid sample and (.) indicates that decimal point must appear somewhere in the field.

9.3.2.1 For quan types CAL, APS, VER, BLK, must be S5.0:1

9.4 For semi-volatile fraction

9.4.1 For quan types CAL and VER, must be NA:NA

9.4.2 Aqueous samples:

9.4.2.1 For quan types APS, PAR, OPR, and BLK: must be 1000:1

9.4.3 Solid samples:

9.4.3.1 For quan types APS, PAR, OPR, and BLK: must be S30.0:1

9.4.3.2 For quan types EPA, FBK: must be S(.)N(.)N(.)N:XXXXX, where S indicates a solid sample and (.) indicates that a decimal point must appear somewhere in the field.

10 Date Extracted

10.1 Must be present for semi-volatile quan report types EPA, BLK, IPR, PAR, and OPR

10.2 Format: MM/DD/YY-X

10.3 Limits: MM=00-12 DD=00-31 YY=83-99

11 -----------> X=Shift: D, S, G

10.4 For semi-volatiles quan report types CAL, VER or for volatiles fraction, must be NA

12 Method

12.1 If present must equal 1624A, 1624B, 1624C, 1625A, 1625B, 1625C

12.2 If fraction V, must be 1624A, 1624B, or 1624C

12.3 If fraction A, B, or C, must be 1625A, 1625B, or 1625C

13 Column length

13.1 If present must be as follows:

13.1.1 If fraction V, must be in range 2.8-3.1 M

13.1.2 If fraction A, B, or C, must be in range 25-35 M

13.2 Unit of measure must be meters (M)

14 Column inside diameter

14.1 If present must be as follows:

14.1.1 If fraction V, must be in range 1-3 mm

14.1.2 If fraction A, B, or C, must be in range 0.2-0.35 mm

14.1.3 Unit of measure must be mm

15 Column initial hold

15.1 If present must be as follows:

15.1.1 Format: Hold @ temp: MM@TTT

15.1.2 Temperature ranges:

15.1.2.1 Volatile: 25-50

15.1.2.2 Semi-volatile: 25-35

15.1.3 Hold ranges:

15.1.3.1 Volatile: 2-4

15.1.3.2 Semi-volatile: 4-6

16 Column temperature program

16.1 If present must be as follows:

16.1.1 Format: initial temp - final temp @ rate: TTT-TTT@RR

16.1.2 Rate must be 8

16.1.3 Initial temp must equal initial hold temp (item 15)

16.1.4 Final temp must equal column final hold temp (item 17)

17 Column final temp/hold

17.1 If present must be as follows:

17.1.1 Format: Hold @ temp: MM@TTT

17.1.2 Temperature ranges:

17.1.2.1 Volatile: 200-250

17.1.2.2 Semi-volatile: 270-290

17.1.3 Hold ranges:

17.1.3.1 Volatile: 5-25

17.1.3.2 Semi-volatile: 10-20

18 Carrier gas flow rate

18.1 If present must be as follows:

18.1.1 Volatile:

18.1.1.1 Range: 20-40

18.1.1.2 Unit of measure: ML/MIN

18.1.2 Semi-volatile:

18.1.2.1 Range: 20-60

18.1.2.2 Unit of measure: CM/S

19 Compound order number

19.1 Must be present

19.2 Range: 001-250

20 Compound number

20.1 Must be present

20.2 Must be valid number in compound table (Table 4)

20.3 Fraction on quan report must agree with number in compound table. If fraction on quan report = C, then table value can be A or B only

20.4 If compound order number = 001, then tabled compound type must be I (internal standard)

21 Mass to charge ratio (M/Z)

21.1 Must be present

21.2 Format: NNN

21.3 Range:

21.3.1 Volatile: 20-250

21.3.2 Semi-volatile: 35-450

21.4 Must equal tabled value for compound (Table 4)

22 Scan number

22.1 Must be present

22.2 Format: NNNN

22.3 Range (retention time/scan):

22.3.1 Volatile: no greater than 3.00

22.3.2 Semi-volatile: no greater than 1.00

22.4 Test performed on internal standard

23 Retention time

23.1 Must be present

23.2 Must be within range of tabled value for compound (Table 50)

24 Reference compound

24.1 Must be present

24.2 Must be numeric

24.3 If compound number is 001-299, 500-699, or 900-1000, reference compound number must be number for an internal standard

24.4 If compound number is 300-429 or 700-829, reference compound number must be:

24.4.1 Compound number minus 100 for numbers in range of 300-499 and 700-799

24.4.2 Compound number minus 300 for numbers in range of 400-429

24.4.3 Compound number minus 400 for numbers in range of 800-829

25 Relative retention time

25.1 Must be present

25.2 Format: MMM:SS M=minutes S=seconds

25.3 Must be within range of table value for the compound (Table 51)

26 Peak area

26.1 Must be present

26.2 Format: NNNNN

26.3 Must be within range of table value for the compound (Table 52)

27 Amount

27.1 Must be present

27.2 Format: NNNNNNN.NNNNN

27.3 Must be within range of tabled value for the compound (Table 53)

27.4 For quan report types EPA, labeled compounds (compound numbers 201-299 and 600-699), amount must be greater than 10 and less than 401.

27.5 For quan report types BLK, amount for all pollutants (compound numbers 001-088, 301-388, 500-599, 700-799, and 900-1000), must be less than 10

28 Unit of measure

28.1 Must be present

28.2 Format:

28.2.1 Volatiles: UG/L

28.2.2 Semi-volatiles: UG/ML

29 Retention time (library)

29.1 Must be present

29.2 Must be within range of tabled value for the compound (Table 50)

29.3 No test on quan report type CAL

30 Relative retention time (library)

30.1 Must be present

30.2 Must be within range of tabled value for the compound (Table 51)

30.3 No test on quan report type CAL

31 Amount (library)

31.1 Must be present

31.2 Must be within range of tabled value for the compound (Table 53)

31.3 No test on quan report type CAL

32 Response factor

32.1 Must be present

32.2 Must be within range of tabled value for the compound (Table 54)

32.3 No test on quan report type CAL

33 Response factor (library)

33.1 Must be present

33.2 Must be within range of tabled value for the compound (Table 54)

33.3 No test on quan report type CAL